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PILOT PRODUCTION AND EVALUATION OF TANTALUM ALLOY SHEET

Prepared by R. L. Ammon and R. T. Begley

SUMMARY PHASE REPORT

Prepared under Navy, Bureau of Naval Weapons

Contract NOw-62-0656-d

APRIL 15, 1962 TO MARCH 15, 1963

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Astronuclear Laboratory
Westinghouse Electric Corporation

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ABSTRACT

Studies of the processing and properties of tantalum rich alloys in the Ta-W-Hf system were carried out. Procedures for the production of pilot quantities of low interstitial T-111 (Ta-8W-2Hf) sheet from 3" diameter ingots were developed, and detailed evaluations of mechanical and physical properties were conducted. The effects of intentional additions of interstitial elements, carbon, oxygen, and nitrogen, on fabricability, weldability, and mechanical properties of T-111 were investigated. Limited amounts of carbon and nitrogen improved mechanical properties while oxygen had little or no effect. Interstitial additions on the order of 0.03 w/o had no adverse effect on fabricability or weldability.

The determination of the optimum composition of T-111 with respect to the substitutional solutes, tungsten and hafnium, consistent with good fabricability, weldability, and high temperature strength is described. Results indicated that a minimum in weld bend transition temperature occurs at a tungsten-hafnium ratio of 4:1, permitting the total solute concentration of T-111 to be raised to 14 w/o without adversely affecting the fabricability and weldability, while resulting in a significant increase in the high temperature tensile properties. Limited studies of the weldability and mechanical properties of Ta-W-Re, Ta-W-Hf-V, and Ta-W-Hf-Re alloys were also carried out.

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INTRODUCTION

This report on the "Pilot Production and Evaluation of Tantalum Alloy Sheet", describes the results of work conducted on Bureau of Naval Weapons Contract NOw-62-0656-d during the period April 15, 1962 to March 15, 1963.

The primary purpose of this program was to conduct detailed studies of the properties and processing of tantalum rich alloys in the Ta-W-Hf system; previous work under Contract NOas-58-852-C had shown that Ta alloys containing 2 to 8 w/o W and 2 to 6 w/o Hf had excellent high temperature properties in addition to good fabricability and very satisfactory low temperature ductility. Of particular interest was an alloy containing Ta-8W-2Hf*. This alloy, designated as T-111, displayed good high temperature properties along with excellent low temperature ductility.

This program involved the complex evaluation of the T-111 alloy and was conducted in the following manner:

1. The production of pilot quantities of low interstitial T-111 sheet from two 3" diameter double arc melted ingots and its evaluation for mechanical and physical properties.

2. The investigation of the effects of intentional additions of interstitial elements carbon, oxygen, and nitrogen on the fabricability, weldability and mechanical properties of T-111.

3. The determination of the optimum composition of T-111 with respect to the substitutional solutes tungsten and hafnium consistent with good fabricability, weldability, and high temperature strength.

In addition to the above-outlined program, several additional Ta-base alloys were investigated on a limited basis to determine their potential as high temperature structural materials.

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*All compositions are given in weight per cent (w/o) unless otherwise indicated.

PROCESSING OF MATERIAL

Sheet material for the evaluation of low interstitial T-111 was produced from two 3" diameter ingots. The processing consisted essentially of double vacuum arc melting 3" diameter ingots, extruding at a 4:1 reduction ratio to rectangular bar, forging to plate, conditioning, recrystallizing, and cold rolling to final gage.

Material for the recrystallization study of T-111, along with interstitial and solute optimization evaluation was produced from non-consumably arc melted button ingots. The buttons were forged to plate, conditioned, recrystallized and cold rolled to sheet.

CONSUMABLE ARC MELTING

Prior to the effective date of this contract, studies of the Ta-8W-2Hf alloy were initiated under Westinghouse sponsorship. Sufficient material was purchased to prepare two 3" diameter ingots, and initial processing and mechanical property data were obtained. More extensive evaluation of this alloy was undertaken with the inception of the contract.

Electrode Preparation. Tantalum plate was obtained from Kennametal, Inc., for fabricating electrodes. Table 1 lists typical analyses of the starting material. First melt electrodes for the heats designated Ta-32 and Ta-34 were made using the sandwich technique. This method consisted of stacking 3/4" wide strips of tantalum, tungsten, and hafnium of the appropriate thickness to give the desired composition and fastening them together to give a rigid composite structure as illustrated in Figure 1. The electrodes were then welded to columbium adapters to facilitate melting. The electrodes for heat Ta-32 were riveted together with pure tantalum rivet stock while electrodes for heat Ta-34 were fusion welded together.

Melting. First melts in each case were made into a 1-7/8" diameter mold using an AC power supply. Melting data are shown in Table 2. AC power was preferred to DC because of the uniform melt-off of material from the electrode, regardless of a large disparity of melting points of the materials involved. The condition of the electrode tips after melting indicated that both tungsten and hafnium were melted off at a uniform rate. Previous experience with tungsten containing columbium alloy electrodes prepared by this technique has indicated that homogeneous ingots free from unmelted tungsten can be produced from single AC melts.[2] Tungsten melts containing Ta and Re have been single AC melted and have been found to be extremely homogeneous.[3]

The first melt ingots were conditioned at the ends to facilitate welding together to form the second melt electrode. A second melt electrode made in this manner is pictured in Figure 1. No material was removed from the diameter.

The second melt of heat Ta-32 was accomplished using a DC power supply with excellent results. However, attempts to melt heat Ta-34 were unsuccessful. The second melt electrode for heat Ta-34 was over twice the length of that for Ta-32 and contained three additional welds which may have contributed to the inability to melt this electrode at current values as high as 4000 amps at 35 volts. After several attempts to DC melt, it was decided to melt the electrode using an AC power supply although the length limitation of the available

TABLE 1 - Chemical Analyses of Starting Material*

Tantalum Supplier - Kennametal, Inc.

Typical Analysis

Carbon	0.012 w/o	Columbium	< 0.01 w/o
Oxygen	0.010 w/o	Titanium	< 0.01 w/o
Nitrogen	0.004 w/o	Iron	< 0.01 w/o

< Silicon 0.01 w/o

Tungsten Suppliers - Fansteel
Kulite Tungsten Co.

Since the tungsten received from these suppliers was in the form of 0.020-inch sheet, it was assumed that the interstitial content of the tungsten was low so as to be of no consequence.

Tungsten Sinter Bar Supplier - Westinghouse, Lamp Division

Carbon 0.0036 w/o Oxygen 0.0010 w/o

Hafnium Supplier - Bettis Atomic Power Laboratory

Carbon 0.0190 w/o Oxygen 0.0030 w/o Nitrogen 0.0010 w/o

Supplier - Wah Chang

Carbon < 0.0030 w/o Oxygen < 0.0030 w/o Nitrogen < 0.0008 w/o

*All values given in weight per cent

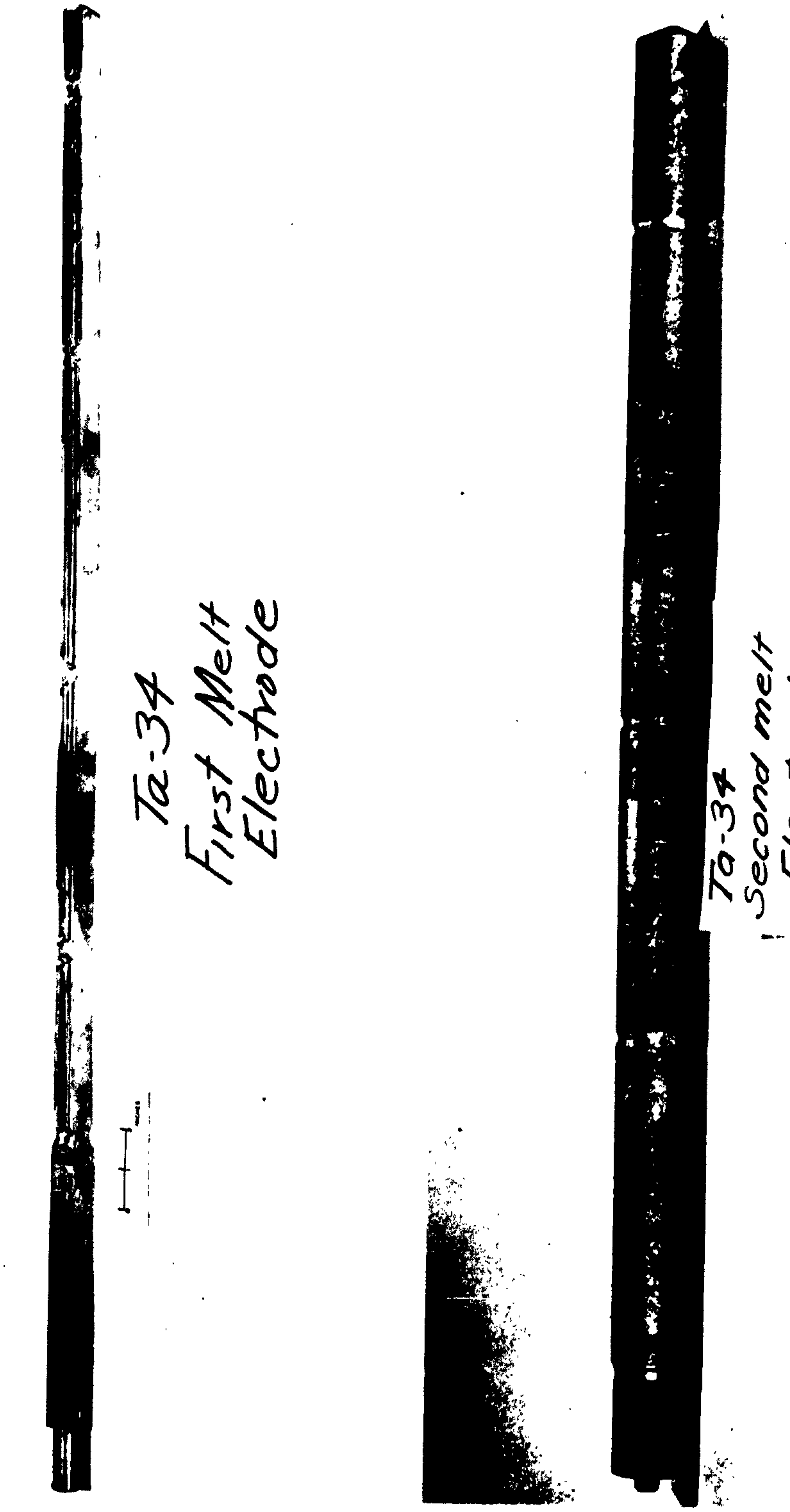

Fig. 1 - First and Second Melt T-111 Electrodes

TABLE 2 - Melting Data

Heat No.	Power Supply	Current (Amps)	Voltage (Volts)	Melt Rate (lbs/min)
Ta-32-1	AC	2880	20-22	3.3
Ta-32-2	AC	2900-3000	22-26	3.3
Ta-32-3	AC	2900	20-23	3.9
Ta-32-4	AC	2900-3000	24-26	3.2
Ta-32 (Second Melt)	DC	3300	30-32	3.3
Ingot weight 26.5 lbs.				
Ta-34-1	AC	2500-3000	28	3.3
Ta-34-2	AC	3000	30	3.2
Ta-34-3	AC	3000	28	3.2
Ta-34-4	AC	2800-3000	25	3.3
Ta-34-A	AC	3000	27	3.2
Ta-34-B	AC	3000	27	3.2
Ta-34-C	AC	3000	28-30	3.3
Ta-34-D	AC	2800-3000	26	2.8
Ta-34 (Second Melt)	DC	4000	35	Did Not Melt
Ta-34-A1	AC	3600	25-35	6.5
Ingot weight 27.5 lbs.				
Ta-34-B2	AC	3600	25-35	6.5
Ingot weight 19.0 lbs.				
Ta-35 (Single Melt)	AC	2700	28	3.0
Ingot weight 6.8 lbs.				

Pressure during all melts was less than 0.5 Torr measured at top of mold.

mold required that two separate melts be made. The results of the AC melts are shown in Figure 2. As can be seen in the photograph, an excellent surface was achieved on both ingots.

PRIMARY AND SECONDARY WORKING

Breakdown of the as-cast ingots was accomplished by extrusion at the Materials Manufacturing Division of Westinghouse. The equipment used was a 700-ton Lowey Hydropress. The die was prepared in accordance with procedures outlined by Tombaugh[4] and plasma sprayed with zirconia. Because of a slight disparity between the diameter of the machined extrusion billet (slightly less than 3") and the I. D. of the extrusion container (3-1/8") a coating of Mo was plasma sprayed onto the O. D. of the billet to prevent upsetting during the extrusion process. In addition, billets Ta-34-A1, which was extruded to a round, and Ta-34-B2, had 1-1/2" Mo nose plugs added to eliminate the unworked nose which is commonly found in extrusions. A picture of Ta-34-A1 is shown in Figure 3. The Mo coating served another useful purpose in preventing contamination of the billet during transfer from the induction furnace to the extrusion press billet container. Sufficient Mo extruded with and remained on the extruded bar so that immediate reheating and forging was accomplished with minimum contamination. Extrusion data are given in Table 3.

The rectangular bar extrusions were forged at 3000°F to 1/2" thick plate. Heat Ta-32 after extrusion and forging is shown in Figure 4. As can be seen in the photograph, some of the Mo coating is evident even after the forging operation. The Mo and contaminated surfaces were removed from the plate by a machining operation. The conditioned pieces were then vacuum annealed as follows:

Ta-32 1-1/2 hrs. at 2750°F
Ta-34-B2 1 hr. at 2700°F

The microstructure of heat Ta-32 in the as-extruded, and the extruded, forged and recrystallized conditions is shown in Figure 5. No evidence of a recrystallization can be seen in the as-extruded material, but after forging and subsequent annealing at 2750°F, a fully recrystallized microstructure was achieved.

TABLE 3 - Extrusion Data

Heat No.	Billet Size	Billet Wt. (lbs)	Extrusion Ratio	Extrusion Size	Temp. (°F)	Load (Tons)
Ta-32	2-13/16" dia. x 4"	16.8	4:1	1-1/4" x 1-3/8" sheet bar	3000	400
Ta-34-A1	2-15/16" dia. x 5-1/8"	20.5	2.5:1	2" dia. round bar	3000	350
Ta-34-B2	2-15/16" dia. x 4-1/8"	16.2	4:1	1-1/4" x 1-3/8" sheet bar	3000	400

Die coated with zirconia according to Tombaugh.[4]
All billets plasma coated with Mo, Ta-34-A1 and Ta-34-B2 each fitted with 1-1/2" thick Mo nose plug. Lubrication used was Fiske 604 on container and die.

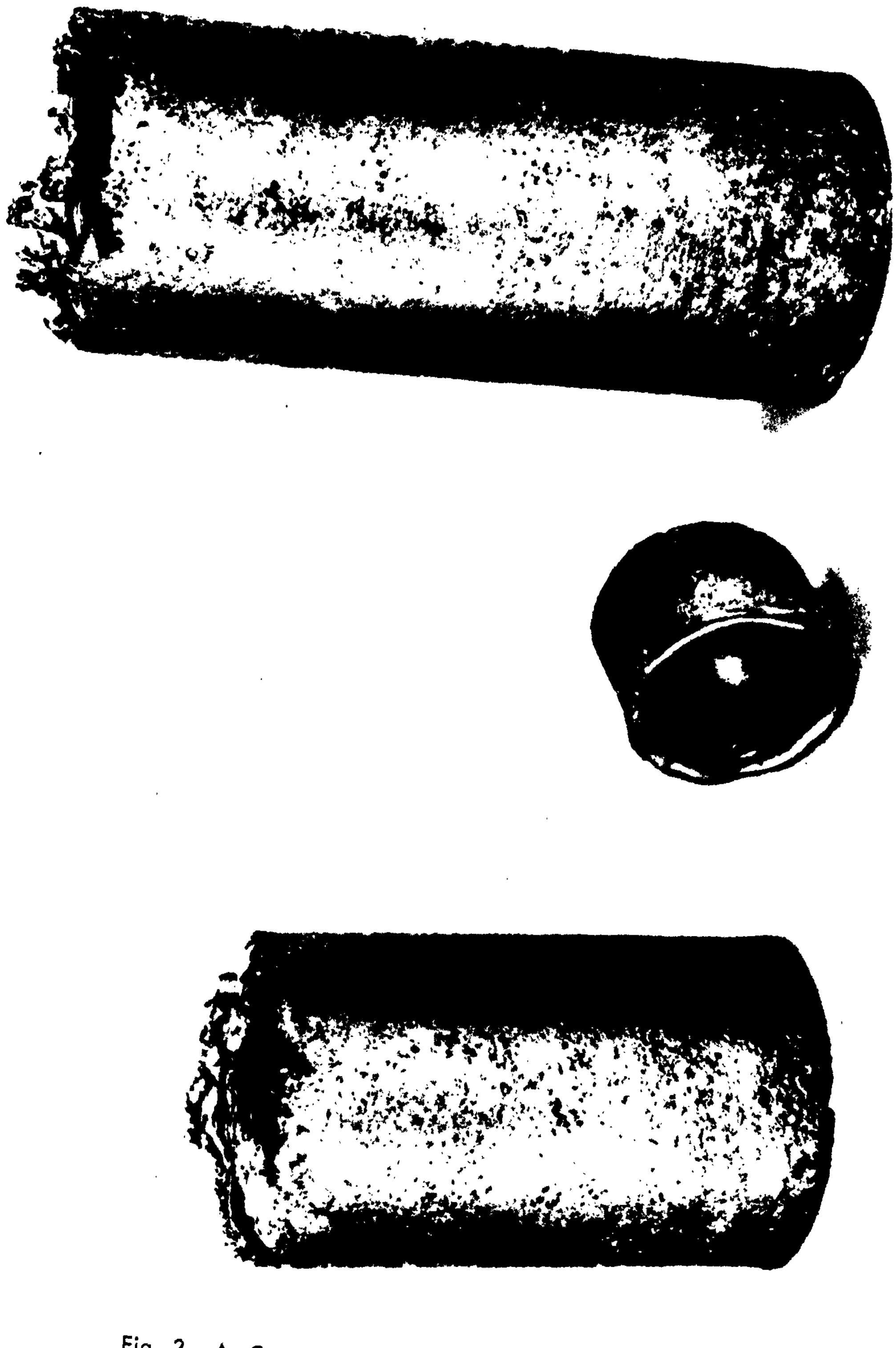

Fig. 2 - As Cast AC Arc Melted T-111 Ingots

Fig. 3 - T-111 Round Bar Extrusion (Ta-34)

Fig. 4 - T-111 Extruded and Forged Sheet Bar (Ta-32)

a) AS EXTRUDED

b) EXTRUDED, FORGED, AND ANNEALED
1 1/2 HRS. AT 2750 °F

Fig. 5 - Microstructure of Low Interstitial T-111 (Ta-32) 100X

Initial rolling of recrystallized Ta-32 was done on a slabbing mill with 18" diameter rolls at a temperature of 500°F, with reductions of 0.040" per pass to a gauge of 0.140". Final rolling was done without reheat on an 8" wide x 8" diameter Stanat laboratory rolling mill. Initial reductions were taken at 30 to 40 per cent with finish reductions of 10 to 15 per cent to a final gauge of 0.025" to 0.028". As a result of the success in cold rolling Ta-32 plate to sheet, Ta-34-B2 plate was cold rolled to final gauge on the 8" x 8" diameter laboratory Stanat mill.

In addition to the two billets that were extruded, a forging blank which consisted of 1-1/2" slice from the Ta-32 billet, was plasma sprayed with Mo and upset forged at 3000°F to plate. Forging was accomplished without reheating and resulted in a reduction of about 80 per cent. The resulting forging is shown in Figure 6. Note the Mo which remained on the upper surface throughout the forging operation. The processing of heats Ta-32 and Ta-34 are summarized in the flow charts of Figures 7 and 8.

Additional low interstitial T-111 sheet was prepared from a single AC arc melted ingot. This heat was designated as Ta-35. The ingot was coated with an Al-Si alloy and side forged to plate at 2200°F. The aluminide coating process has been found to be quite satisfactory for preventing contamination of Ta-W-Hf alloys[5]. The as-forged plate can be seen in Figure 9. The plate was conditioned by shaping to remove the coating and any possible contaminated material on the surface. The slab was subsequently given a recrystallization anneal for one hour at 3000°F.

NON-CONSUMABLE ARC MELTING

The evaluation of the effect of interstitial additions on the properties of T-111 and the optimization of the solute composition of T-111 were conducted using sheet material produced from small button ingots. The investigation of other Ta-base alloys was also conducted using similar sheet material.

Melting. Button ingots were made using the same starting materials as shown in Table 1. Carefully weighed out compacts of the desired compositions were placed in the individual molds of a multi-hearth furnace. The chamber was evacuated to a pressure of at least 5×10^{-5} Torr, back-filled with high purity argon and evacuated a second time to 5×10^{-5} Torr. The chamber was back-filled to 1/3 of an atmosphere with a mixture of helium and argon in the ratio of 4 to 1. The buttons were melted using a conventional water-cooled tungsten electrode. The ingots, which were approximately 1/2" thick by 3" in diameter, were melted several times on each side, sectioned and examined for signs of segregation. The sections were remelted several more times to form homogenous ingots. A typical as-melted button ingot is shown in Figure 10.

Primary and Secondary Working. Breakdown of the as-cast button ingots was accomplished by forging at 2200°F. To protect the ingots from oxidation during the heating cycle and forging operation, an aluminide coating was applied to each ingot[5]. The ingots were forged to approximately 1/4" thick, a reduction of about 50%. Five typical as-forged button ingots are shown in Figure 11. The forged plates were conditioned by removing 0.020" to 0.040" from each surface. The conditioned plates were given a recrystallization annealed for one hour at 3000°F in vacuum. The recrystallized slab was cold rolled on a laboratory size

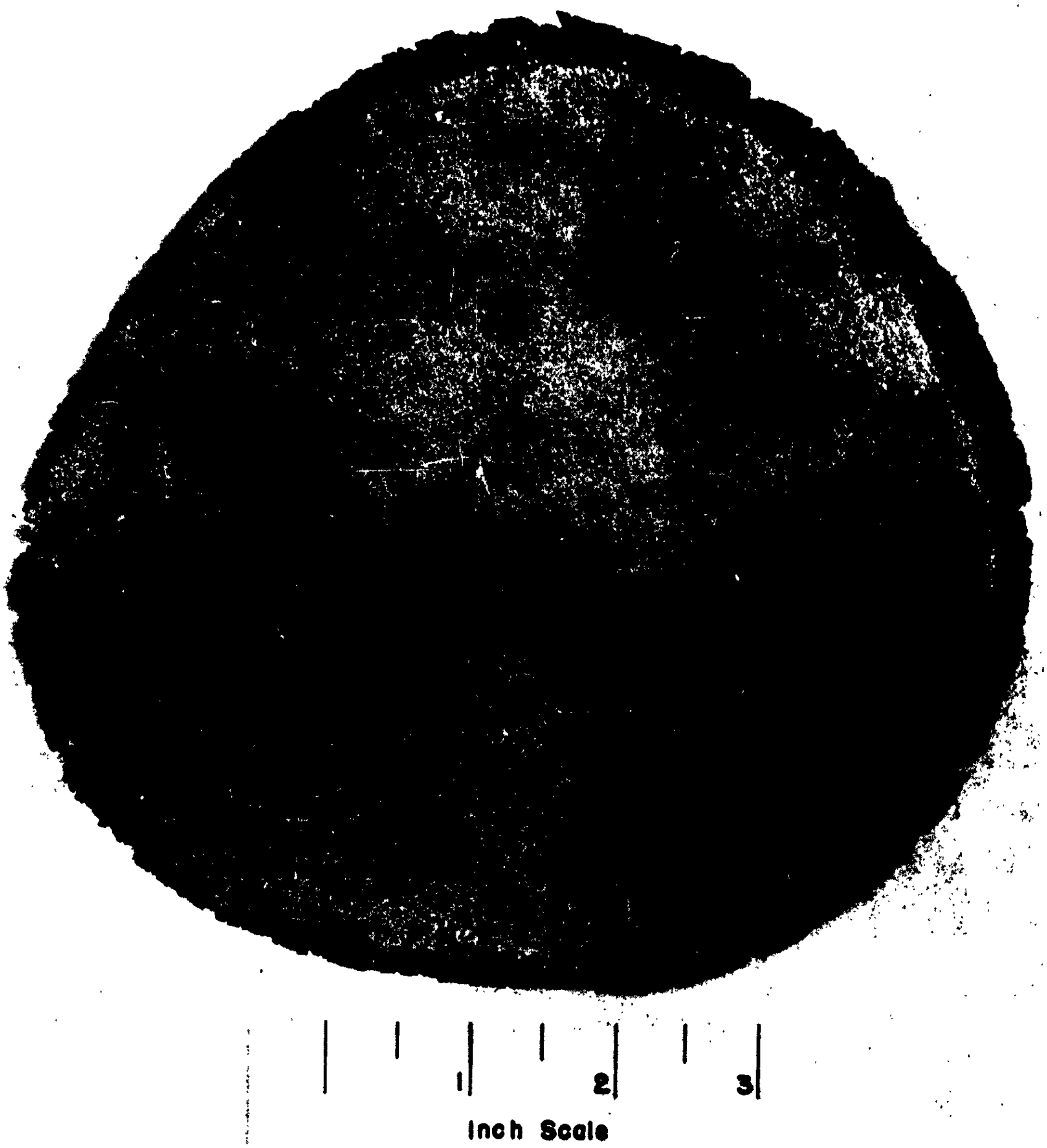

Fig. 6 - T-111 Upset Forged Plate (Ta-32)

Heat Ta-32

Four - 8 lb. electrodes constructed
using "sandwich" technique

First melts AC arc melted
into 1-7/8" dia. mold

Ingots machined and welded to
form second melt electrode

Second melt DC arc melted into
3" dia. mold
Conditioned billet wt. 26.5 lbs.

Extrusion Billet
2.8" dia. x 4.0" (16.8 lbs)
Plasma coated with 1/16" Mo

Extruded at 3000°F to
Sheet bar 1-3/8" x 1-1/4" x 16"

Reheat to 3000°F and
forged to 1/2" plate

Material cut and conditioned to slabs
0.293" x 1.8" x 6.4"
0.336" x 1.9" x 5.7"
0.205" x 3.0" x 4.8"

Slabs - annealed 1-1/2 hours
at 2750°F

Rolled on 18" dia. slab mill
at 0.040" reductions to
0.140"

Final rolling done on a
8" x 8" dia. Stanat mill
at reductions of 10 to 15%
to final gauge 0.025 to 0.028"

Yield 7.0 lbs. sheet
6" x 20" x 0.025"
5.7" x 22" x 0.028"
4.8" x 22" x 0.028"

Forging Blank
2.8" dia. x 1.56" (5.8 lbs)
Plasma coated with 1/16" Mo

Upset forged at 3000°F
to 1/2" plate

Material conditioned to slab
0.138" x 2.2" x 5.7"

Slab annealed 1-1/2 hr. at 2750°F

Cold rolled to 0.050" sheet

Fig. 7 - Flow Diagram for the Processing of Heat Ta-32

Heat Ta-34

Eight - 7-1/2 lb. electrodes constructed
using "sandwich" technique

First melt made into 1-7/8" dia. mold
using AC power supply

Ingots cropped and welded to form
second melt electrode

Three unsuccessful attempts to DC melt into a
3" dia. mold resulted in the loss of 7 lbs. of material

Second melt AC arc melted into 3" dia. mold
Two ingots produced - Ta-34A1 - 26.5 lbs.
Ta-34B2 - 19.0 lbs.

Ta-34-A1

Extrusion Billet - 5-1/8" x 2.94" dia. -
20.5 lbs. Mo plasma coated 0.03" thick
1-1/2" Mo nose plug

Extruded at 3000°F to 1-7/8" dia. round

Extrusion ratio 2.5:1

Ta-34-B2

Extrusion Billet - 4-1/8" x 2.9" dia. -
16.2 lbs. Mo plasma coated 0.03" thick
1-1/2" Mo nose plug

Extruded at 3000°F to bar
1-3/8" x 1-1/4" x 17"

Reheated to 3000°F and Forged
1/2" plate

Plate cut and conditioned to 3 slabs
4-1/8" x 1-5/8" x 0.446"
5-1/2" x 2.0" x 0.425"
3-3/4" x 3-1/8" x 0.488"
(1/3 lost due to
secondary pipe)

Slabs annealed 1 hr/2700°F

Cold rolled at 10% per pass to 0.045".
This sheet was sent to Watertown
Arsenal for evaluation.

Fig. 8 - Flow Diagram for the Processing of Heat Ta-34

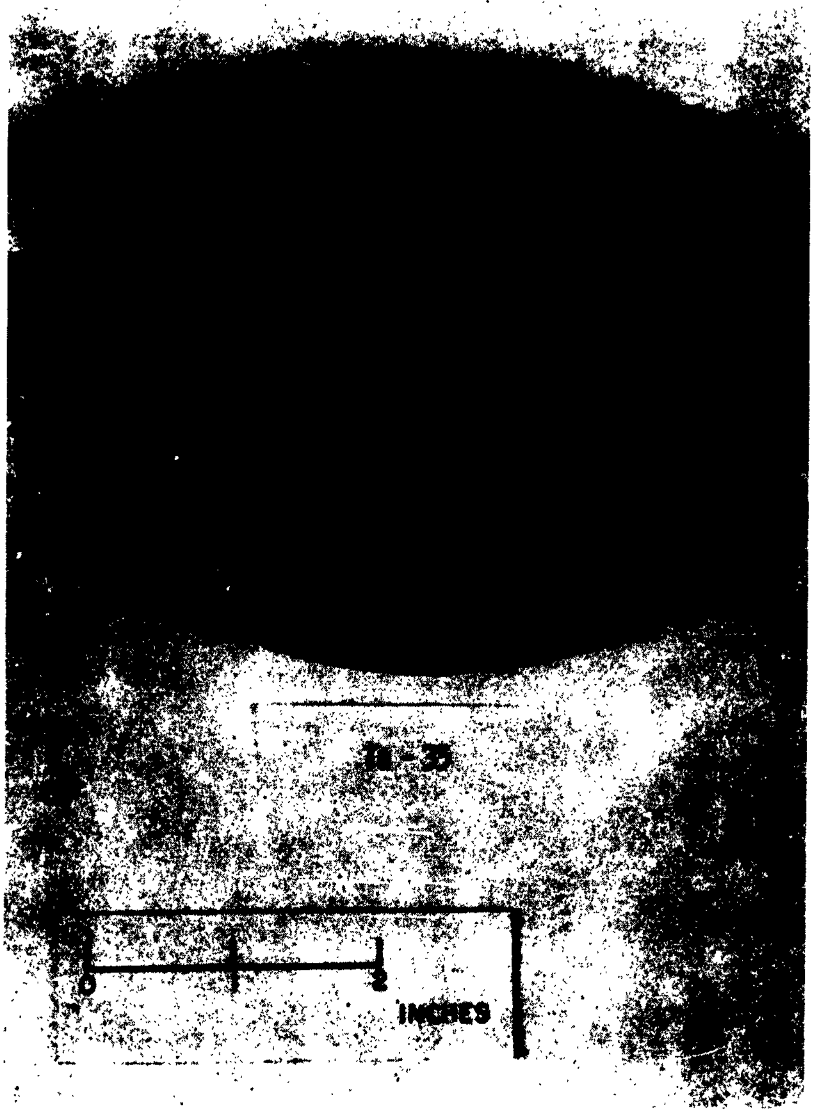

Fig. 9 - T-111 Side Forged Ingot (Ta-35)

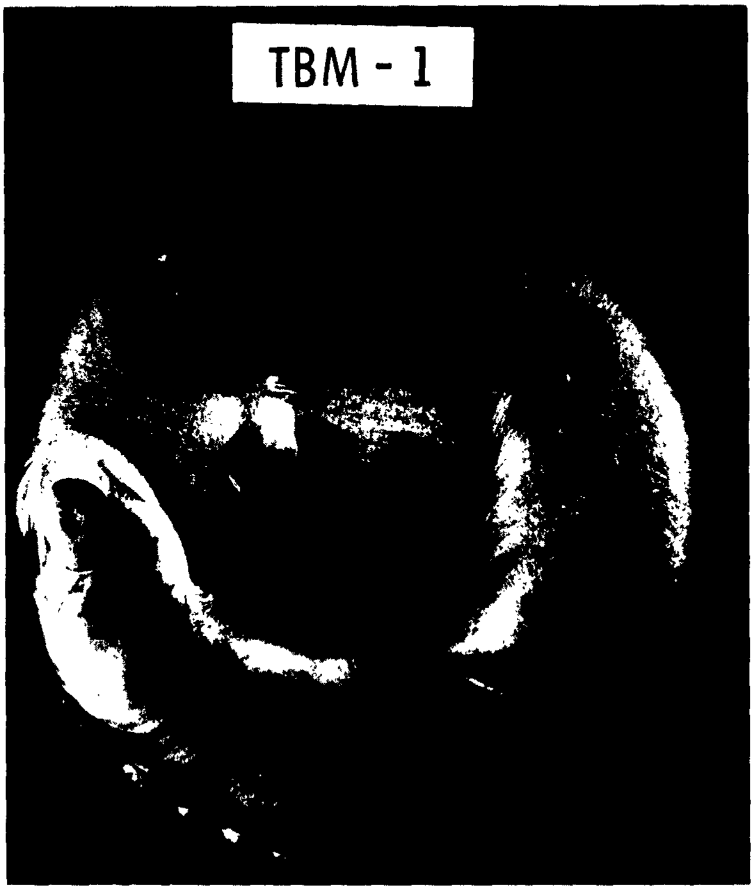

Fig. 10 - Typical As Cast Button Ingot

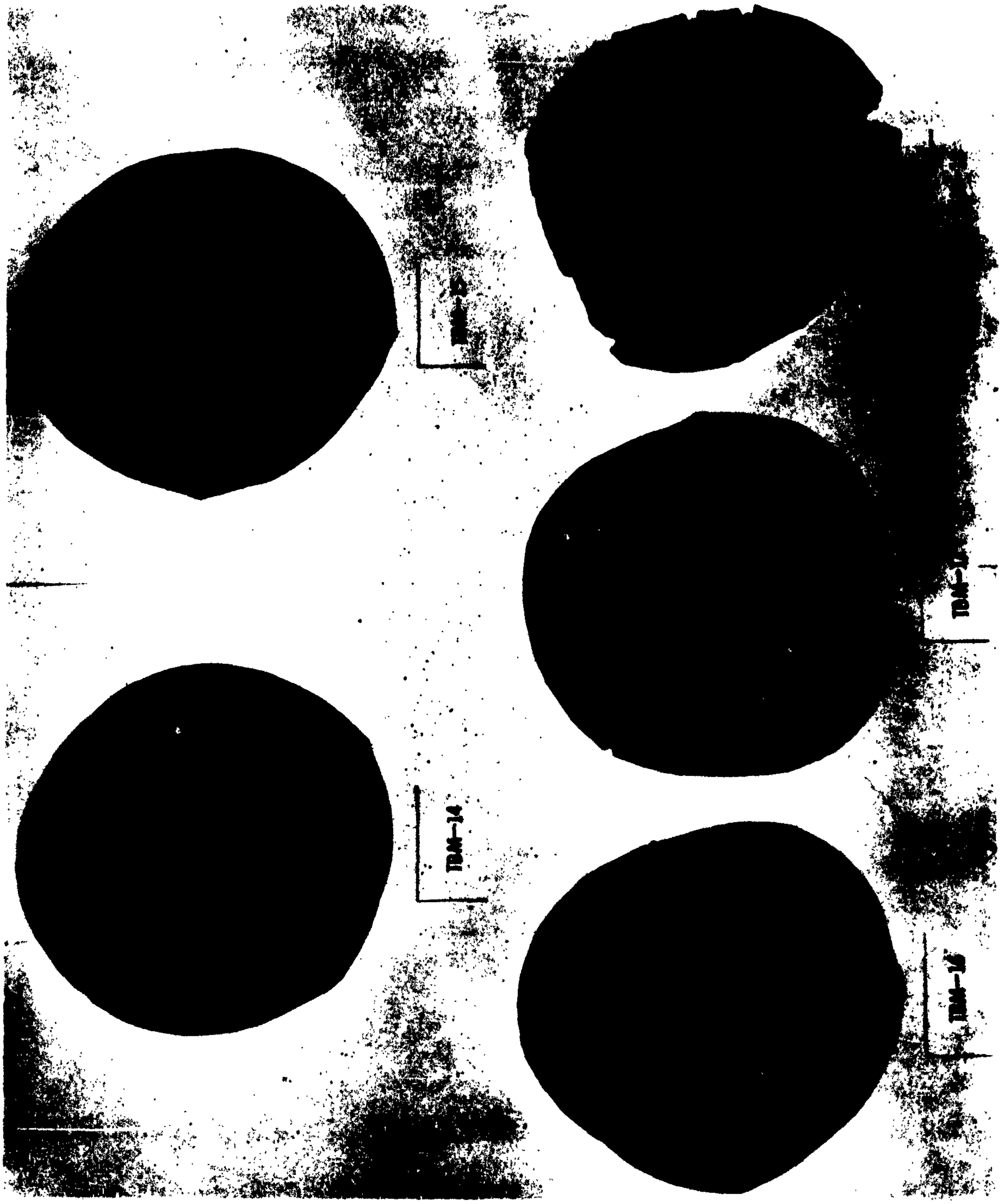

Fig. 11 - Typical As Forged Button Ingots

Stanat rolling mill. Reduction per pass was on the order of 10 per cent. The quality of the cold rolled sheet material was generally very good as illustrated in Figure 12.

BASELINE DATA FOR T-111

Processed heats Ta-32, Ta-34 and Ta-35 provided pilot quantities of sheet for this phase of the T-111 evaluation. Chemical analyses of these heats are listed in Table 4. Approximately two square feet of 0.020" sheet from heat Ta-32 was sent to Battelle, as directed by the Bureau of Naval Weapons, for inclusion in an Air Force coating program. Approximately two square feet of 0.045" sheet was also forwarded to Watertown Arsenal for evaluation of mechanical properties.

MATERIAL AND EXPERIMENTAL PROCEDURE

Baseline data for T-111 were obtained using material produced from 3" diameter ingots as described earlier in this report. All low temperature tensile testing was conducted at the Westinghouse Research Laboratories. High temperature tensile testing was conducted by the Metcut Research Associates, Inc., of Cincinnati, Ohio. Test procedures recommended by the MAB were followed where possible[6]. Tensile tests were performed using sheet specimens having a 1" gauge length and a 0.25" gauge width. Figure 13 shows the specimen geometry that was used. Specimen thickness was generally 0.05"; however, some specimens 0.028" thick were also tested. Subatmospheric test temperatures were achieved using the technique of Wessel[7]. Strain rate for test temperatures up to 800°F was generally 0.005 in/in/min. Some initial tests were conducted at a strain rate of 0.06 in/in/min. Elevated temperature tests were conducted in vacuum at pressures generally below 5×10^{-5} Torr. Strain rate for the high temperature tests was 0.05 in/in/min.

All heat treatments were conducted in vacuum at pressures below 5×10^{-5} Torr. Samples were wrapped in Ta foil prior to annealing to minimize contamination.

TABLE 4 - Chemical Analyses of T-111 Heats

Heat No.	Chemical Analyses (w/o)				
	W	Hf	O	N	C
Ta-25	7.86	1.97	0.0100	0.0068	0.0026
Ta-32	7.50	1.75	0.0018	0.0039	0.0012
Ta-34	7.70	2.53	0.0009	0.0004	0.0011
Ta-35	8.08	2.50	0.0014	0.0013	0.0066

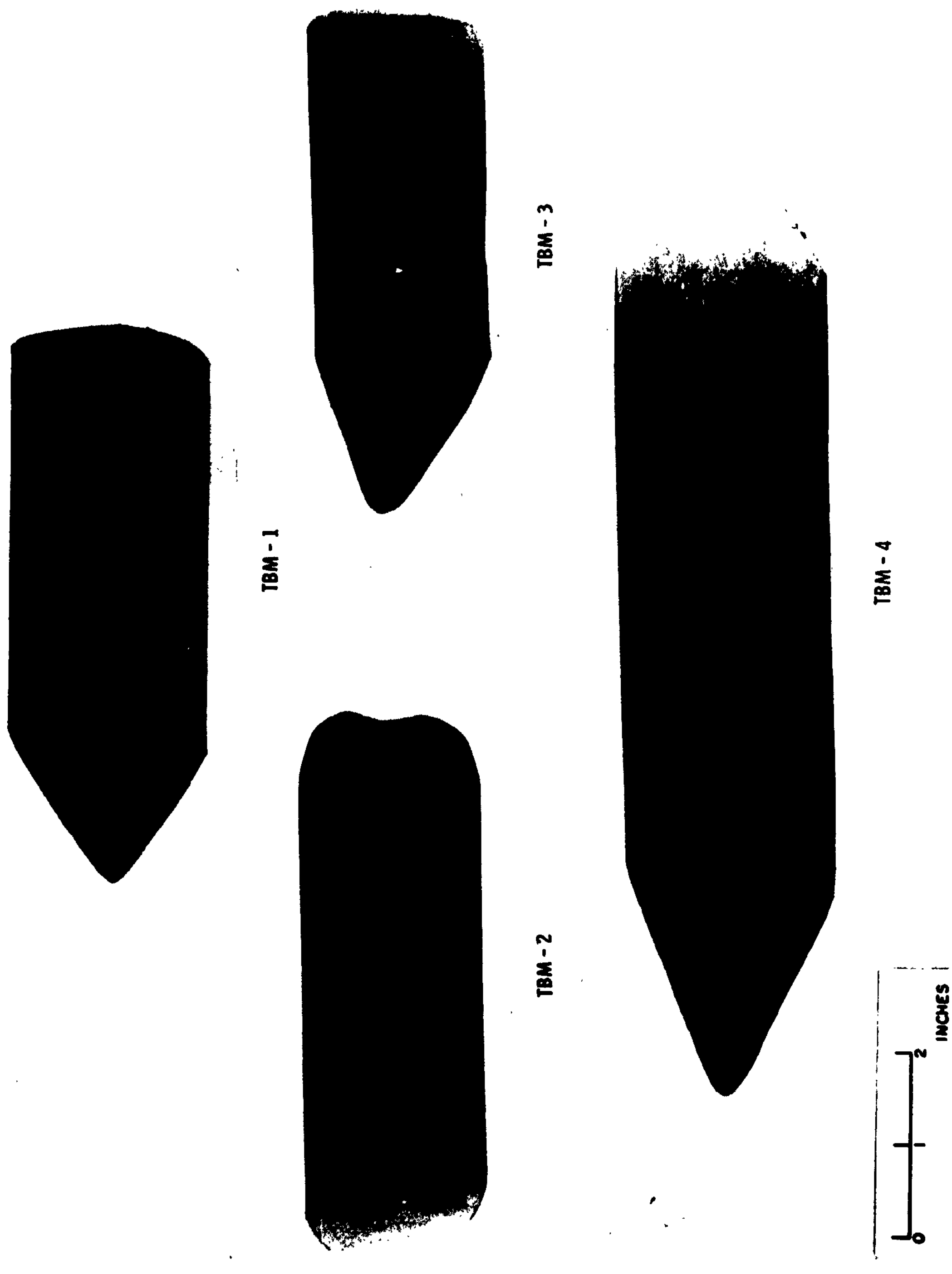

Fig. 12 - Typical As Rolled Ta-W-Hf Sheet

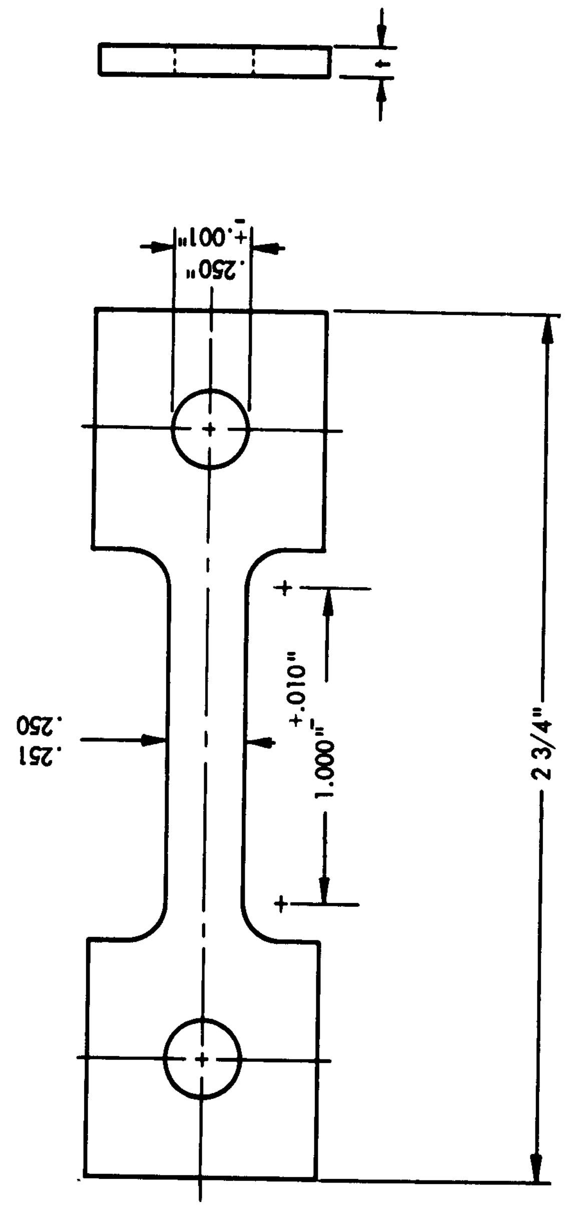

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Fig. 13 - High Temperature Tensile Specimen

Mechanical Properties

Base-line mechanical property evaluation for T-111 included tensile property determinations in the temperature range -452 to 3500°F, notch tensile tests at room temperature and -320°F, and stress rupture tests at 2400°F.

Low Temperature Tensile Properties. Tensile data were obtained for T-111 at temperatures ranging from -452 to 800°F for recrystallized and stress-relieved material. The results are listed in Table 5 and plotted as a function of temperature in Figure 14. Also included are results for heat Ta-25, which was tested as part of a previous program.[1] As can be seen from Figure 14, the results for heat Ta-34 are somewhat lower than those of heat Ta-32. Although these heats were processed in a similar manner, several factors may account for the difference in properties. As shown in Table 4, Ta-32 contained approximately twice the interstitial element concentration as did heat Ta-34. The tensile specimens for heat Ta-32 material were machined from 0.028" thick sheet having a cold rolled surface, while specimens from heat Ta-34 were 0.050" thick and were surface ground to final thickness. These minor differences in composition, specimen geometry and preparation may account for the apparent difference in tensile properties.

Tensile tests were conducted at -452°F on stress-relieved and recrystallized T-111 to explore the very low temperature deformation behavior of this alloy. The specimen in the stress-relieved condition exhibited only 0.3% total elongation at fracture. However, the fracture was of the ductile shear type and the reduction of area at fracture was of the order of 40%. The low total elongation value merely reflects the fact the deformation was localized in the fracture area, which is typical of Cb and Ta tested at cryogenic temperatures. The recrystallized specimen exhibited considerly higher elongation (~17%). Pronounced discontinuous yielding was observed, as shown in the stress-strain curve of Figure 15. Serrated stress-strain curves have been observed in Ta and other bcc metals at low temperatures, as well as in fcc and cph metals.[19] A number of explanations have been proposed for the observed discontinuous yielding. One possibility is that deformations occur in twin bursts. However, no evidence of twinning was observed in the tensile specimen after completion of the test. A more likely explanation in this particular instance is extremely localized heating as a result of deformation, as discussed by Bechtold, Wessel, and France.[20] It is of interest to note that T-111, like pure tantalum shows no evidence of a transition from ductile to brittle behavior at temperatures as low as -452°F.

In order to determine the effect of specimen orientation or tensile properties, and to provide data on recovery phenomena in T-111, room temperature tensile tests were conducted on material annealed one hour at temperatures ranging from 2000 to 3000°F. The material, heat Ta-32, was cold rolled to 90% reduction prior to annealing. Data were obtained on sheet material, 0.028" thick, tested with the specimen axis perpendicular, parallel, and at forty-five degrees to the rolling direction of the sheet. Tensile data are listed in Table 6 and plotted as a function of prior annealing temperature in Figure 16. The data for material tested in the as-rolled condition indicate no gross anisotropy. However, material annealed at 2200°F indicate some influence of rolling directionality on yield and ultimate strength properties. The material annealed at 2600°F showed essentially no dependency of tensile properties on test orientation with respect to rolling direction. This behavior, no doubt, can be associated with the propensity of columbium, tantalum, and their alloys to initiate recovery

TABLE 5 - Low Temperature Tensile Properties of T-111 Sheet

Heat	Temperature (^{o}F)	Ultimate Tensile Strength (ksi)	Yield Strength 0.2% Offset (ksi)	Elongation (%)	Strain Rate (in/in/min)	Remarks
Ta-25	-320	190.0	184.0	18	0.06	Material reduced and stress relieved 1 hr. at 2000°F
	-100	150.0	146.0	17	0.06	
	75	135.0	130.0	15	0.06	
Ta-32	-452	-	232.2	0.3	0.005	Material reduced 90% and stress relieved 1 hr. at 2000°F
	-320	194.5	188.5	12.6	0.005	
	-100*	-	157.7	-	0.005	
	75	150.0	144.8	9	0.005	
	75	151.3	148.2	10	0.06	
	400	124.0	121.6	4	0.005	*Temperature control valve malfunctioned
	800	114.8	111.6	3.6	0.005	
	-452	187.7	-	17.5	0.005	Material reduced 90% and recrystallized 1 hr. at 3000°F
	-320	150.6	145.2	27	0.005	
	-100	108.8	93.8	24	0.005	
	75	90.4	82.5	29	0.005	
	75	93.1	87.9	28	0.06	
	400	68.0	57.0	23	0.005	
	800	57.2	43.2	15.5	0.005	
Ta-34	-320	147.5	131.0	31	0.005	Material reduced 80% and recrystallized 1 hr. at 3000°F
	75	84.0	68.6	31	0.005	

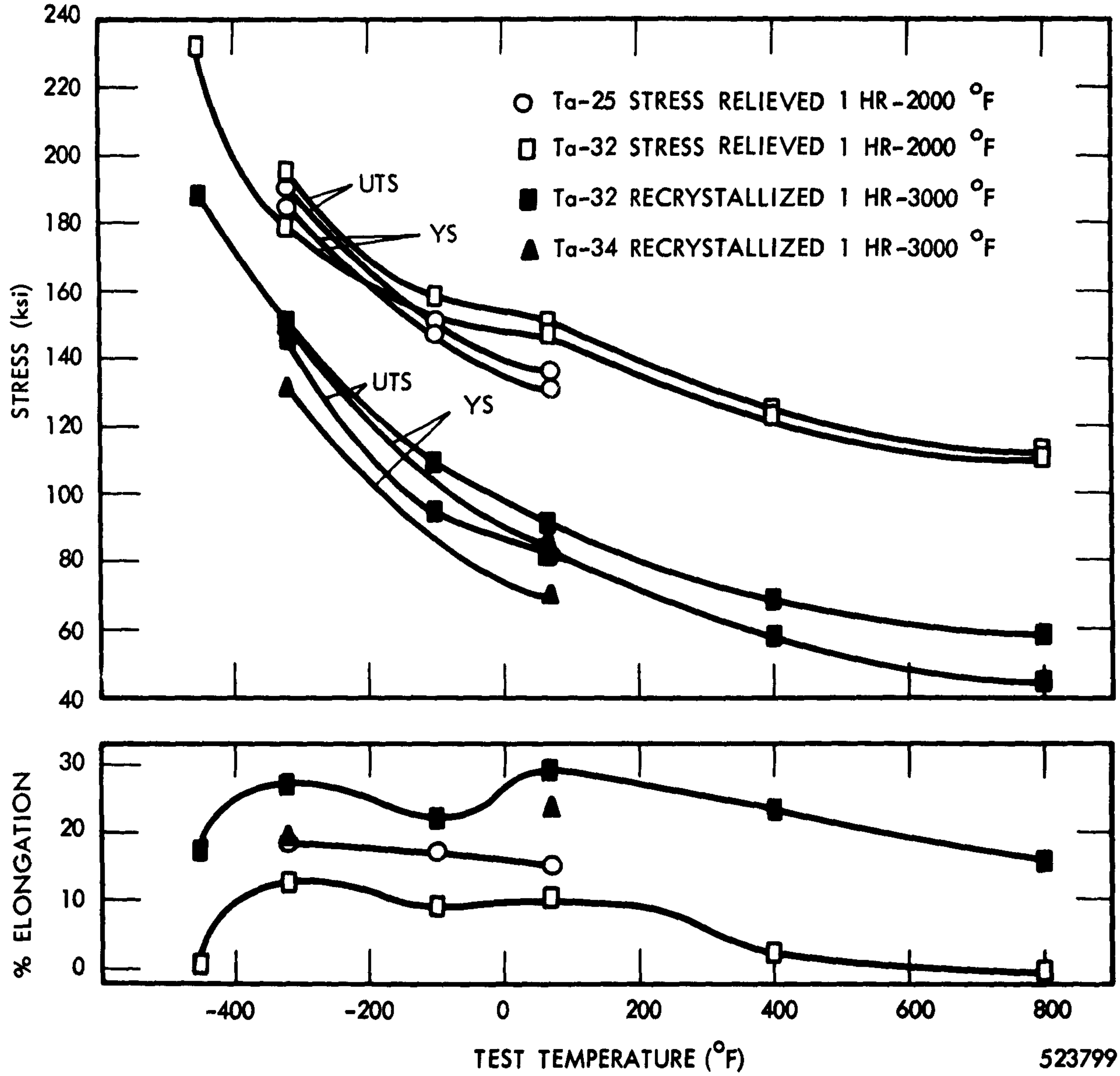

Fig. 14 - Low Temperature Tensile Properties of T-111 Sheet

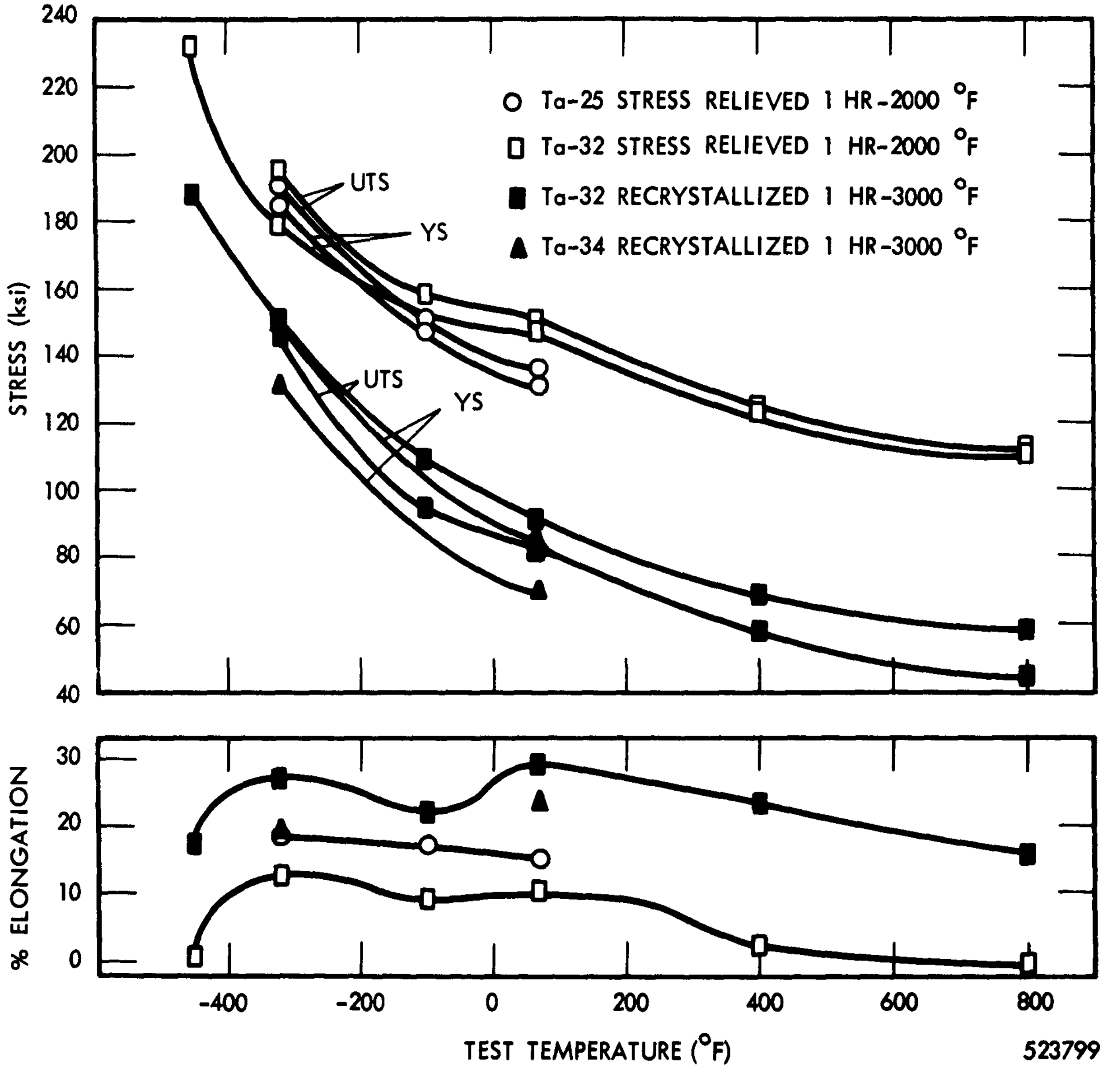

Fig. 14 - Low Temperature Tensile Properties of T-111 Sheet

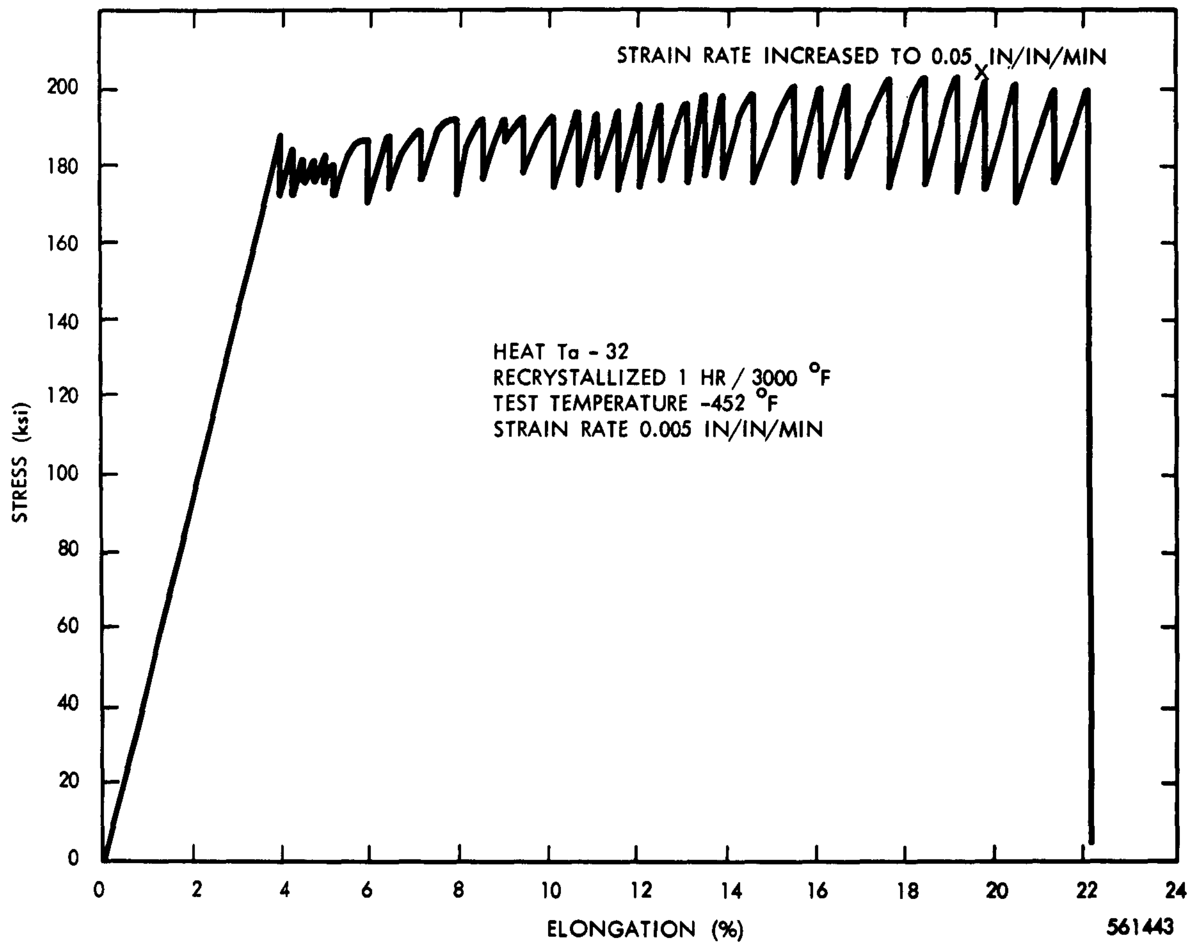

Fig. 15 - Load-Elongation Curve for Recrystallized T-111 Tested at $-452^{\circ}F$

TABLE 6 - Room Temperature Tensile Properties of T-111 Sheet after a 1 Hr. Anneal at Various Elevated Temperatures

Annealing Temperature (1 Hr.)	Ultimate Tensile Strength (ksi)	Yield Strength 0.2% Offset (ksi)	Elongation (%)	Remarks
As rolled	-	158.0	1	Tested perpendicular to rolling direction
2200	139.4	132.9	7	
2400	131.4	125.0	11	
2600	95.7	91.9	26	
2900	98.0	93.7	28	
As rolled	167.2	159.6	4	Tested parallel to rolling direction
2000	150.3	144.8	9	
2200	131.0	125.0	13	
2400	122.0	115.0	17	
2600	91.3	89.9	29	
2900	91.9	90.4	31	
3200	99.8	87.4	28	
As rolled	165.5	156.2	5	Tested 45° to rolling direction
2200	126.0	124.7	15	
2400	113.5	109.6	18	
2600	92.4	89.6	30	

Strain rate 0.06 in/in/min
Material cold rolled 90%

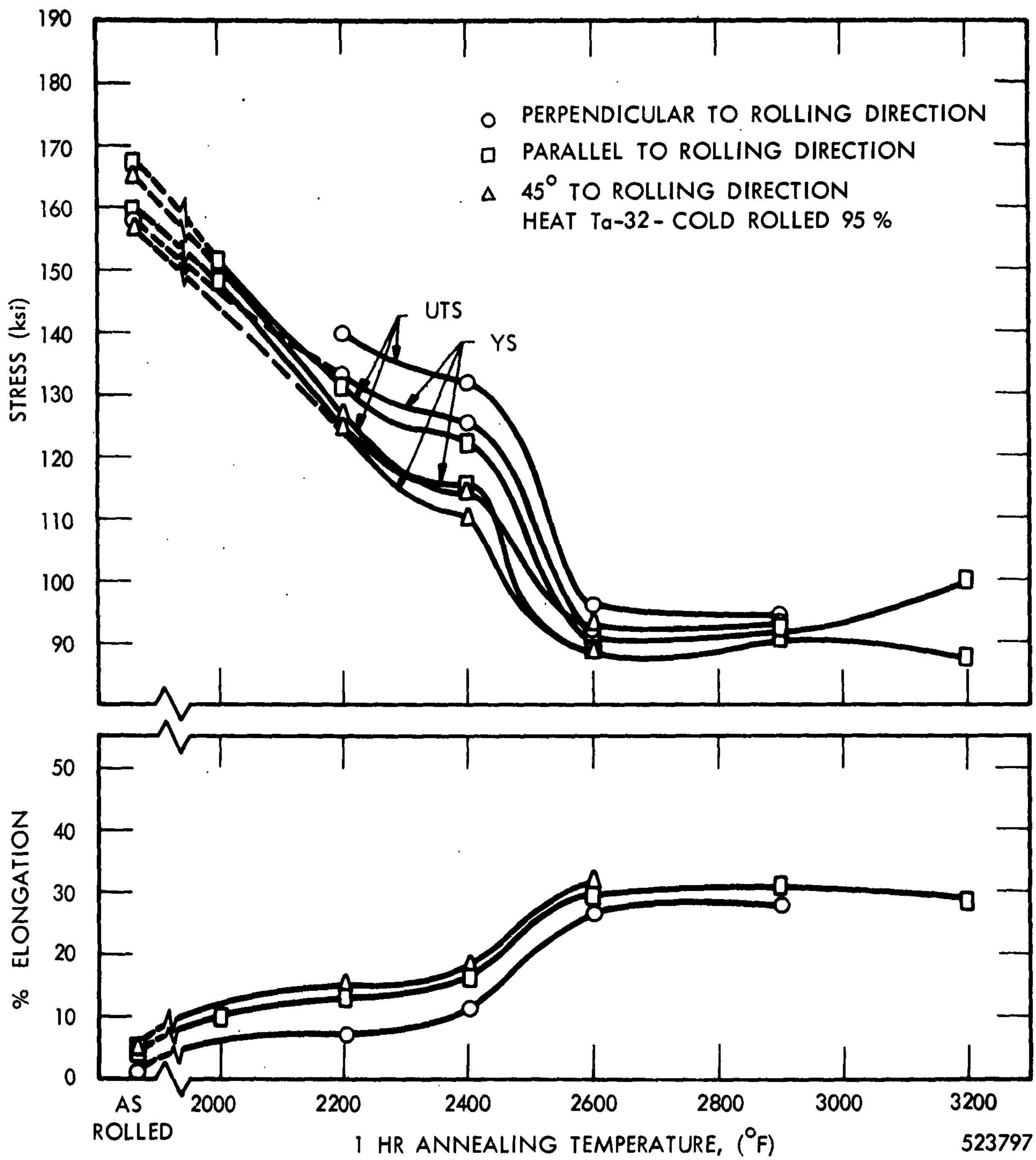

Fig. 16 - The Effect of Annealing Temperature on the Room Iemperature Tensile Properties of T-111 Sheet

at relatively low temperatures and to essentially completely recover ductility prior to the onset of recrystallization.

The tensile fractures of the as-rolled sheet specimens were of the shear type in all cases. This evidence indicates that this alloy is not sensitive to the forty-five degree brittleness which has been encountered in other refractory metals and alloys.

Notched Tensile Properties. Notched tensile tests at room temperature and -320°F were performed using sheet specimens obtained from heat Ta-35. Specimen geometry is shown in Figure 17. Data are listed in Table 7.

TABLE 7 - Notched Tensile Properties of T-111 Sheet

Temperature (°F)	Ultimate Strength (ksi)	Yield Strength 0.2% Offset (ksi)	Elongation (%)	Reduction in Area (%)	Remarks
75	96.85		7	82	Notched
75	85.2	75.4	40	70	Unnotched
-320	157.7		5	32	Notched
-320	136.5	127.4	34	67	Unnotched

Strain Rate - 0.005 in/in/min
Kt = 3.3*

The sheet material was reduced 90% by cold rolling and given a recrystallization anneal, one hour at 3000°F. The notched - unnotched ratio was 1.15 for both test temperatures, using a notch geometry to give a Kt of 3.3*. Work at Battelle has shown the Ta-10W-alloy to be notch insensitive at temperatures as low as -420°F.[9] However, the tests were conducted using round bars with a stress concentration factor Kt of approximately 3. The Ta-10W had a notched - unnotched strength ratio of 1.5 for both stress relieved and recrystallized material. The notched - unnotched ratio for the T-111 was 1.15 as compared to the ratio of 1.5 tor Ta-10W. The difference in these values is probably more attributable to the difference in specimen geometry than to the intrinsic difference in the alloys. Under similar conditions of specimen geometry, T-111 would most likely show notch properties similar to the Ta-10W alloy.

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*Attempts were made to produce a specimen with a Kt of 6, as recommended by the MAB. However, it was not possible to produce a sufficiently small root radius using conventional machining techniques.

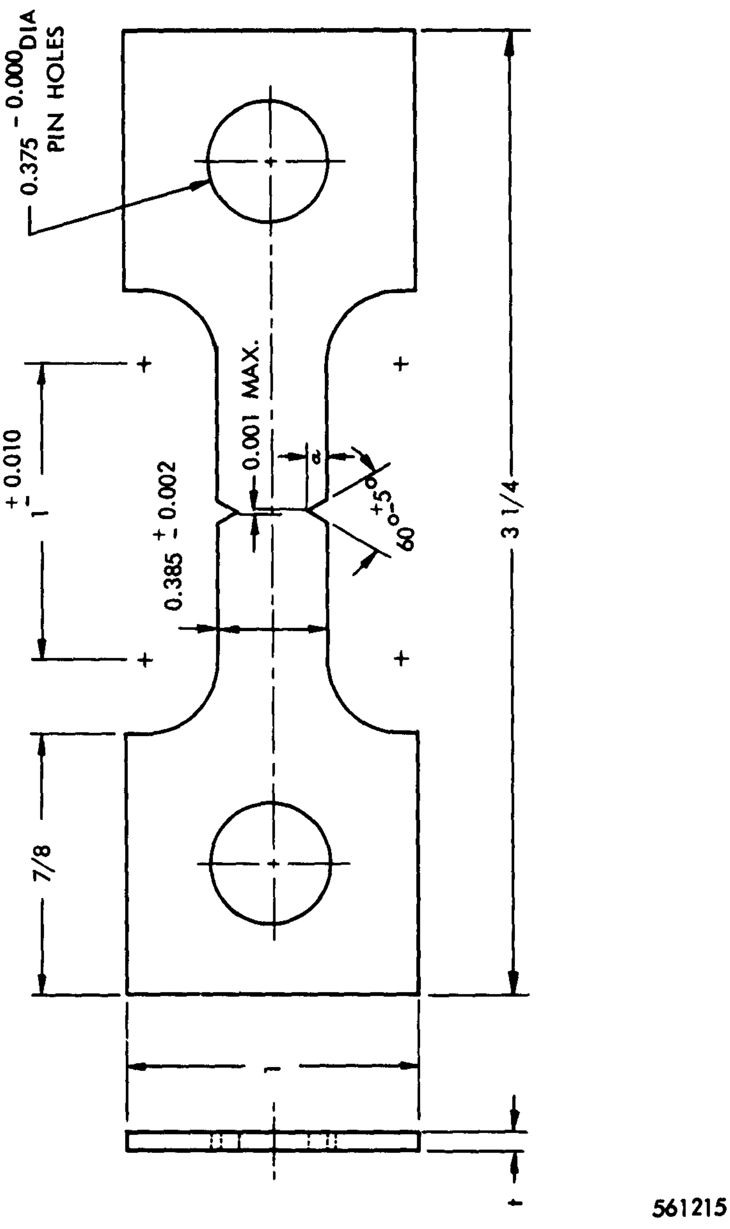


Fig. 17 - Notched Tensile Specimen

Elevated Temperature Tensile Properties. Table 8 lists elevated temperature data for several heats of T-111 alloy sheet. The data for heat Ta-25 have been previously reported with the exception of the 3000°F properties for recrystallized material[1]. Data are presented for heat Ta-32 for material in the wrought-stress relieved and recrystallized conditions. Material from heat Ta-34 was tested in the recrystallized condition only. Figures 18 and 19 show the yield and ultimate strengths of the various T-111 heats as a function of temperature. The data for material tested in the wrought-stress relieved condition represent material with different mechanical and thermal histories. Heat Ta-25 was warm rolled at 2200°F from as-extruded sheet bar stock. Heat Ta-32 was cold rolled from recrystallized sheet bar stock and received 90% reduction. A specimen from heat Ta-32, which was tested at 2400°F, was cold rolled 65% following a recrystallization anneal. The data for this test were the higher of the two 2400°F results for wrought-stress relieved material of heat Ta-32. The significance of these results will be pointed out later in this report.

The effect of specimen geometry, interstitial element content, and prior mechanical and thermal history seems to diminish at 2700°F and above. This temperature was above the recrystallization temperature for this material. The data for the recrystallized material from the low interstitial heats Ta-32 and Ta-34 were consistent throughout the elevated temperature range.

The ductility data show high elongation at fracture for material tested at 2700°F in the wrought-stress relieved condition. The abnormally high elongation value was due to the fine grain size produced by recrystallization during heating to test temperature. Since the test temperature was just above the recrystallization temperature for this material, a fine grain size was produced which was apparently able to accommodate more plastic strain than the larger grain size material. As test temperature was raised, longer heating times, as well as higher temperature, promoted grain growth, thus resulting in a return to normal ductility values as shown by the elongation curve (Figure 19) for materials tested well above the recrystallization temperature. Somewhat similar effects of grain size on elevated temperature ductility have been observed in columbium alloys[8].

Stress Rupture Properties of T-111. Stress rupture tests were conducted on T-111 specimens at 2400°F in vacuum at pressures below 5×10^{-5} Torr. Heat Ta-32 was tested in the wrought-stress relieved condition, and Ta-34 in the fully recrystallized condition. The results are listed in Table 9. Metallographic examination of the stress rupture bars tested in the stress relieved condition revealed a partially recrystallized microstructure in the sample having the shortest rupture life. The specimen having the intermediate rupture time was 80-90% recrystallized, while the test bar which exhibited the longest rupture life (27 hours) was completely recrystallized. Under these conditions of temperature and stress, recrystallization was initiated and proceeded as the test time increased. The recrystallized samples from heat Ta-34 exhibited somewhat higher rupture times and lower creep rates at equivalent stress levels than the stress relieved material. The difference in rupture strength is not attributed to any difference in chemical composition between the two heats (Table 4), but rather to the occurrence of recrystallization during testing of the stress relieved material. The occurrence of recrystallization during creep testing has a significant effect in increasing creep rate and lowering rupture life with respect to recrystallized material. Similar effects have been observed in columbium[10].

TABLE 8 - Elevated Temperature Tensile Properties of T-111 Sheet

Heat	Temperature (°F)	Ultimate Tensile Strength (ksi)	Yield Strength 0.2% Offset (ksi)	Elongation (%)	Strain Rate (in/in/min)	Remarks
Ta-25	2200	85.0	78.0	15	0.06	Warm rolled 95%
	2500	54.0	38.8	26	0.06	Stress relieved 1 hr.
	2700	29.0	23.7	64	0.06	at 2000°F
	3000	20.5	19.5	57	0.06	Specimens 0.050" thick
	3000	17.2	17.2	46	0.05	Recrystallized 1 hr. at 3000°F
Ta-32	2000	92.1	67.5	8	0.05	Cold rolled 90%
	2200	67.1	52.2	20	0.05	Stress relieved 1 hr.
	2400	42.4	38.6	28	0.05	at 2000°F
	*2400	50.7	43.8	26	0.05	Specimens 0.028" thick
	2700	25.4	21.0	76	0.05	
	3000	16.3	14.1	52	0.05	
	3500	11.3	10.9	43	0.05	
	2000	61.1	34.9	18	0.05	Cold rolled 90%
	2200	49.3	28.6	25	0.05	Recrystallized 1 hr.
	2400	37.3	23.5	36	0.05	at 3000°F
	2700	30.9	24.4	30	0.05	Specimens 0.028" thick
	3000	14.8	11.9	48	0.05	
	3500	13.0	12.6	34	0.05	

*Cold rolled 65% - 0.050" sheet specimen

(continued)

TABLE 8 - Elevated Temperature Tensile Properties of T-111 Sheet

(continued)

Heat	Temperature (^{o}F)	Ultimate Tensile Strength (ksi)	Yield Strength 0.2% Offset (ksi)	Elongation (%)	Strain Rate (in/in/min)	Remarks
Ta-34	2000	53.4	30.0	23	0.05	Cold rolled 82%
	2200	45.0	25.3	27	0.05	Recrystallized 1 hr.
	2400	38.0	26.4	29	0.05	at $3000^{o}F$
	2400	39.0	27.0	10	0.15	Sheet specimen 0.050"
	2700	29.0	23.0	31	0.05	thick
	2700	30.5	22.7	23	0.15	
	3000	15.6	14.5	31	0.05	
	3500	8.75	8.5	52	0.05	

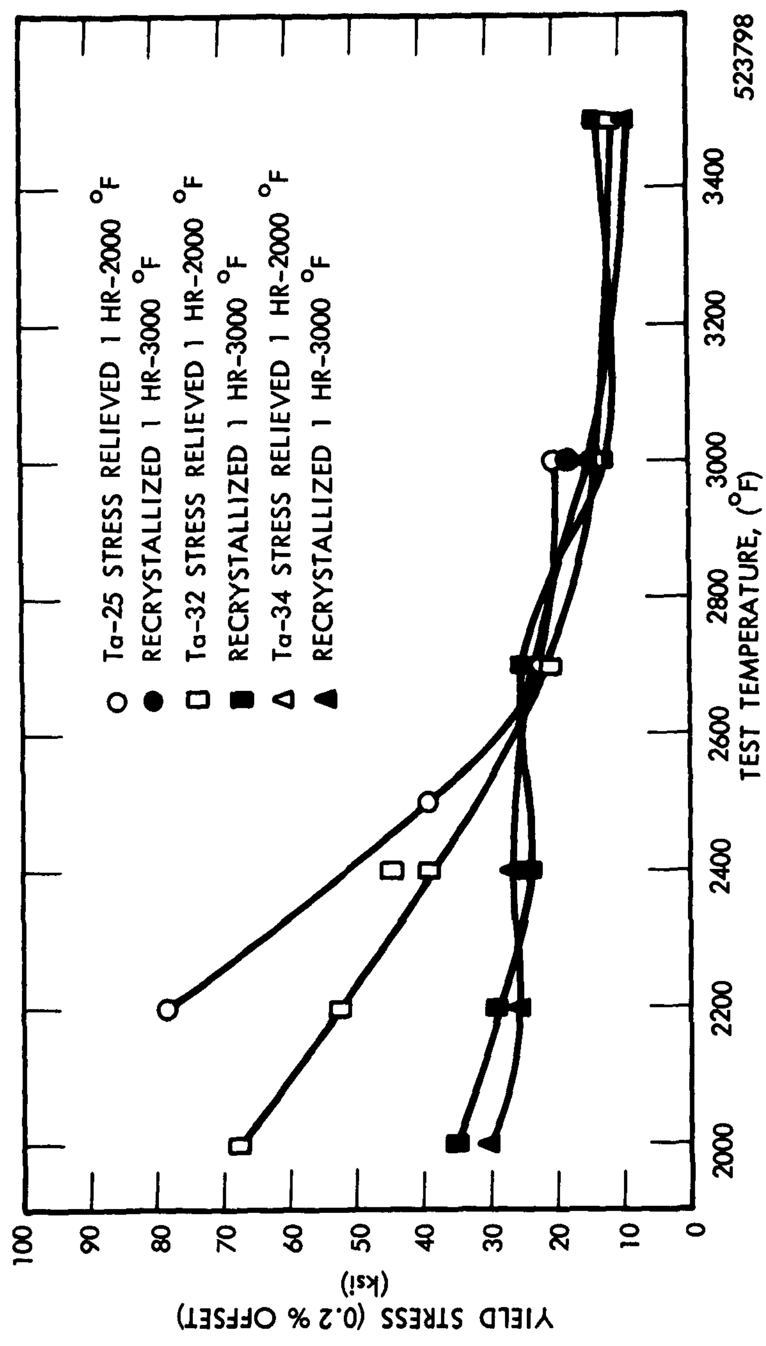


Fig. 18 - The Yield Strength of T-111 Sheet at Elevated Temperatures

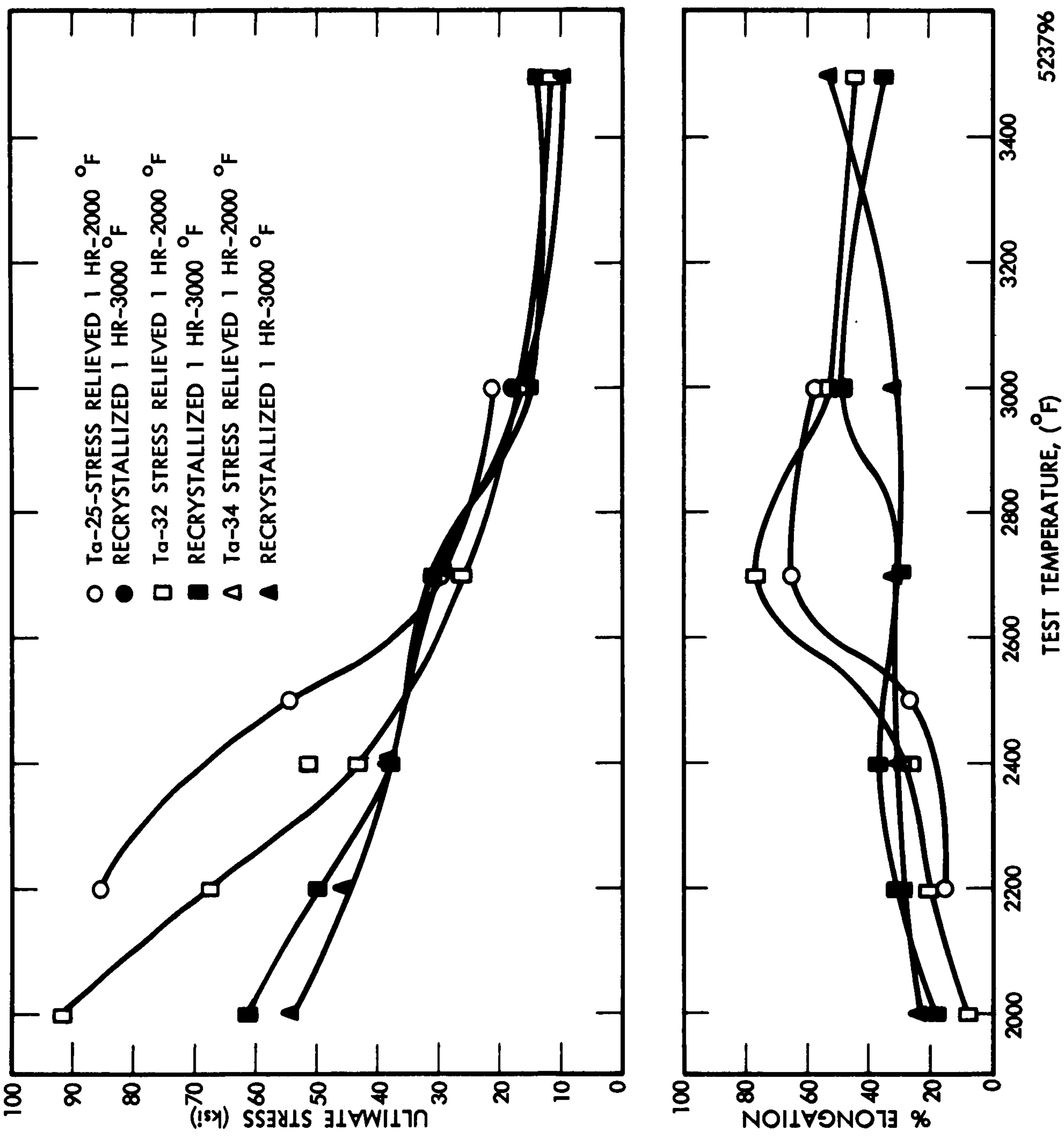


Fig. 19 - Tensile Properties of T-111 Sheet at Elevated Temperatures

In addition to the higher rupture life of the recrystallized material a significant difference was noted in strain at fracture. The recrystallized material shows a lower total elongation than the stress relieved material at both stress levels. The obvious conclusion which can be drawn from these results is that the process of recrystallization during creep allows material to accommodate greater plastic strain than initially recrystallized material.

Chemical analyses of the gauge section of specimens which were tested at the lower stress levels and consequently for longer times revealed an increase in oxygen content. The test conditions of 5×10^{-5} Torr for long time tests at elevated temperatures was apparently not sufficient to prevent gross oxygen contamination by the specimens when procedures developed for short time tensile testing were employed. It was found that when leak-rate and ultimate pressure requirements were made more stringent, along with wrapping the gauge of the test specimen with Ta foil, contamination was drastically reduced. For this reason the 13.2 hour rupture life at a 25,000 psi stress was considered to be the more appropriate value for low interstitial T-111 under the given conditions. As a result of this finding all subsequent stress rupture tests were conducted using the more stringent vacuum technique and wrapping of the gauge with Ta foil was adopted as standard procedure.

TABLE 9 - Stress Rupture Properties of T-111 Sheet Tested at 2400°F

Heat No.	Stress (ksi)	Minimum Creep Rate (%/hr)	Transition Time (hrs)	Rupture Time (hrs)	Elongation (%)	Remarks
Ta-34	30	5.87	2.44	3.0	30	Material reduced
	25	1.28	2.25	7.3	40	80%. Recrystallized
	25	0.34	12.0	27.0	21	1 hr. at 3000°F
	25	1.30	5.9	13.2	43	
Ta-32	30	12.50	1.0	2.3	56	Material reduced
	25	7.09	2.0	4.3	58	65%. Stress relieved
	20	0.98	8.2	25.7	94	1 hr. at 2000°F

Weld Characteristics

To evaluate the weld characteristics of low interstitial T-111, weld bend tests were conducted on sheet from heat Ta-34 to determine the transition temperature for 2T and 4T* bend radii. Aging studies of welded material were also conducted using stress relieved and recrystallized base metal.

Experimental Procedure. Bend specimen for weldability evaluation were made from butt welded sheet. TIG butt welds were performed in a weld chamber designed by W. N. Platte and described in other reports.[11] Welding was done automatically after the

*T denotes sheet thickness.

chamber was vacuum purged and back-filled with argon. The sheet stock which ranged from 0.050" to 0.057" in thickness, was rigidly held in a copper clamping device designed to give high restraint while providing a heat sink for rapid cooling of the welded material.

Weld Bend Ductility. Two types of specimens were used for this evaluation. One type, which was 0.055" thick by 1/4" wide by 1-1/8" long with the weld perpendicular to the longitudinal direction, was used initially. Since this type specimen allows for maximum utilization of available material, its use in other parts of this program was imperative. To provide a basis for comparison, the bend transition temperature for T-111 was determined using specimens of the above-mentioned geometry. Specimens of stress relieved and recrystallized base material were tested in the as-welded condition. No surface preparation or post weld anneals were used before bend testing. Bend specimens in both conditions were tested at -320°F at a deflection rate of 1" per minute. 2T and 4T bend conditions were imposed on the specimen with the top of the weld which ran parallel to the bend axis in tension. At -320°F welded T-111 sheet with the base metal in either the stress relieved or recrystallized condition was completely ductile over 2T and 4T bend radii.

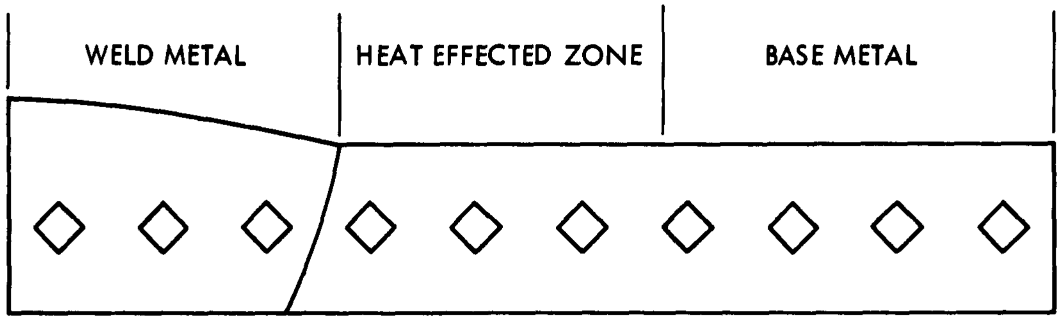
The second bend specimen type that was used was 12T wide by 24T long with the weld running parallel to the long dimension. This size specimen conforms to the recommendation of the MAB. Using identical test conditions as described above, weld bend specimens of the second type as prescribed by the MAB were tested at -320°F at a deflection rate of 1" per minute. The results showed that T-111 was completely ductile over 2T and 4T bend radii.

Weld Aging Study. An aging study was conducted on welded low interstitial T-111 sheet to determine the stability of the base metal, fusion and heat affected zones after one hour aging treatments at temperatures ranging from 1200 to 2200°F. The aged welds were examined metallographically and hardness surveys were conducted to evaluate the effect of the anneals. Strips were welded using the procedures described in the previous section. One weld was made of stress relieved base metal, the other was of recrystallized material. Specimens were cut perpendicular to the weld direction. These specimens were cleaned and etched then wrapped in Ta foil and annealed in vacuum for one hour. The results of the hardness survey are shown in Figures 20 and 21.

The hardness surveys show no gross hardness variations for either condition after aging at all temperatures. The study shows that no second phase appears in the weld metal or heat affected zone after aging for one hour at temperatures up to 2200°F. These results are encouraging since the production of stable welds is a primary prerequisite for many applications.

Correlation of Hot Hardness with Tensile Properties for T-111

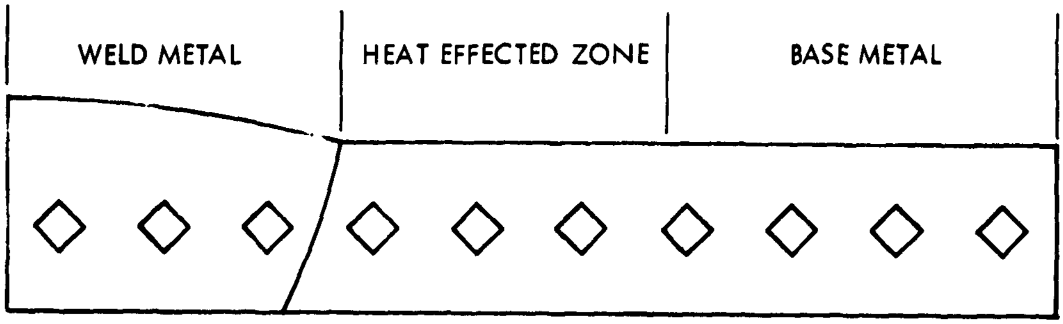
Begley, et al[8], have demonstrated a good correlation between hot hardness and short time tensile strength for columbium and columbium base alloys at temperatures up to 2200°F. Since hot hardness measurements are an extremely useful means of establishing the effect of compositional and thermal treatment variations on elevated temperature properties, experimental work was carried out to establish the correlation between hot hardness and tensile properties for tantalum base alloys. Hot hardness data were obtained on two heats of T-111 and pure tantalum for comparison with tensile properties at corresponding temperatures. Hot hardness samples were obtained from the same sheet stock which had previously been used to establish short time tensile properties. Hot hardness data were obtained for material in the stress



HARDNESS TRAVERSE (VHN)

Aged 1 Hr										
As Welded	227	221	228	213	219	218	224	224		
1200°F	218	228	219	227	213	219	216	216		
1400°F	227	216	227	230	219	225	228	216	219	
1600°F	228	227	221	216	224	222	228	219	218	213
1800°F	238	228	225	221	221	230	224	227	224	
2000°F	225	216	222	218	219	215	215	216	219	222
2200°F	224	224	221	221	218	224	222	224	216	221

Fig. 20 - Aging Study of Welded Recrystallized Low Interstitial T-111 Base Metal



HARDNESS TRAVERSE (VHN)

Aged 1 Hr										
As Welded	225	222	219	221	230	235	297	304	299	
1200°F	227	222	213	215	225	233	302	304		
1400°F	222	232	225	228	222	227	297	309	306	
1600°F	228	230	227	222	221	222	254	306	312	309
1800°F	230	225	218	218	225	251	312	306	304	
2000°F	236	227	222	219	219	224	281	304	309	312
2200°F	215	222	212	221	216	222	260	279	279	285

Fig. 21 - Aging Study of Welded Stress-Relieved Low Interstitial T-111 Base Metal

relieved and fully recrystallized conditions. Tests were carried out in a vacuum hot hardness machine constructed at the Westinghouse Research Laboratories[1]. Hardness impressions were made with a sapphire pyramid indenter at pressures below 5 x 10^{-5} Torr. At least 4 impressions were made at each temperature for each alloy.

The correlation between hot hardness and tensile properties established for T-111 and Ta is shown in Figure 22. Hardness and tensile properties of a particular alloy or type of alloy are functions of a number of complex variables including rate of strain hardening and strain rate sensitivity. This being the case, the relationship between hardness and tensile strength would be expected to be peculiar to the particular metal or alloy being considered. It is interesting to note in Figure 22 that the ultimate strength-hardness value correlations for tantalum alloys at 2000, 2200, and 2400°F agree quite well with the relationship established for columbium alloys. The yield strength-hardness values, however, do not agree. As pointed out by Tabor[12], indentation hardness measurements are essentially a measure of the yield stress of the material being examined, hence, the most meaningful correlation of hot hardness is with yield strength.

Tensile strength-hardness values for stress-relieved material are not included in Figure 22. Room temperature hardness measurements made before and after hot hardness testing indicated that additional recovery was occurring during the heating cycle and causing erratic results.

Recrystallization Behavior

In order to study the recrystallization behavior of the T-111 alloy, a non-consumably arc melted button ingot was used. The button heat was prepared using the techniques described earlier in this report. The ingot, TBM-6, contained 0.0021 w/o oxygen, 0.0081 w/o nitrogen, and 0.0086 w/o carbon. The ingot, which was 1/2" thick by 3" in diameter, was forged to 50% reduction in thickness at 2200°F to provide 1/4" thick plate. The forged plate was then conditioned by a shaping operation. The material was recrystallized one hour at 3000°F and then cold rolled to reductions corresponding to 25, 40, 50, 60, 75, and 90% reduction in area. Material was removed at each stage of reduction. Samples were taken and annealed for one hour at temperatures ranging from 2000 to 3000°F in 200°F increments. The samples were mounted for metallographic examination so that the sheet thickness parallel to the rolling direction was the surface examined. Metallographic examination was used to determine the degree of recrystallization. Hardnesses were also measured and are shown as a function of annealing temperature in Figure 23. The data for the 40 and 60% cold worked material were omitted for the sake of clarity. The hardness for the 95% cold worked material was taken from sheet produced from heat Ta-32. The interstitial content of Ta-32 material was considerably lower than the sheet produced from the button ingot. This difference in interstitial content probably accounts for the difference in behavior of the 90 and 95% reduced materials after comparable high temperature heat treatments.

After one hour at 2600°F metallographic examination of the 75% worked material indicated that recrystallization was just beginning. The 90% worked was partially recrystallized while 95% worked material showed an essentially completely recrystallized structure. As expected, the material which received lesser amounts of cold work required

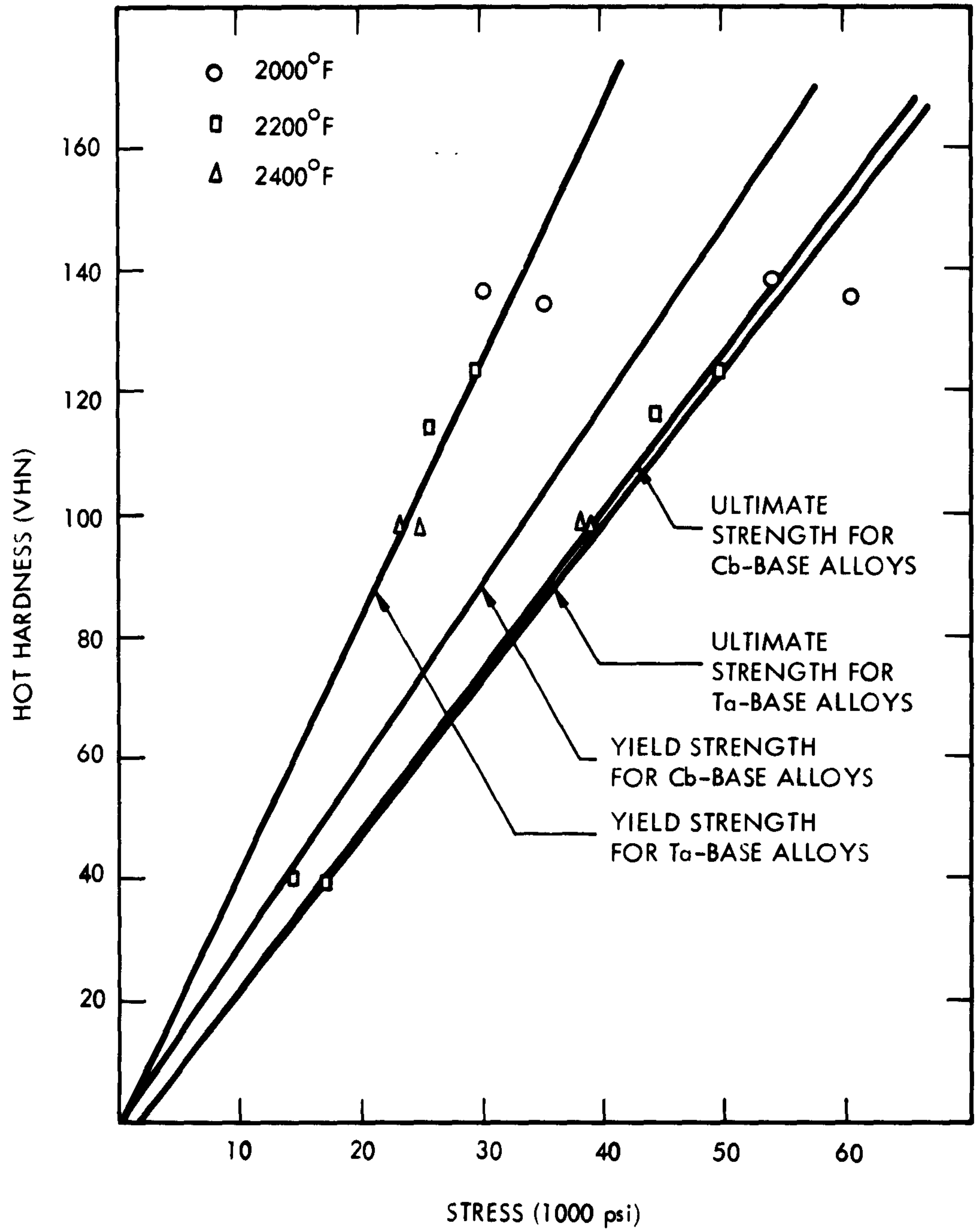


Fig. 22 - Correlation of Hot Hardness with Tensile Strength for T-111

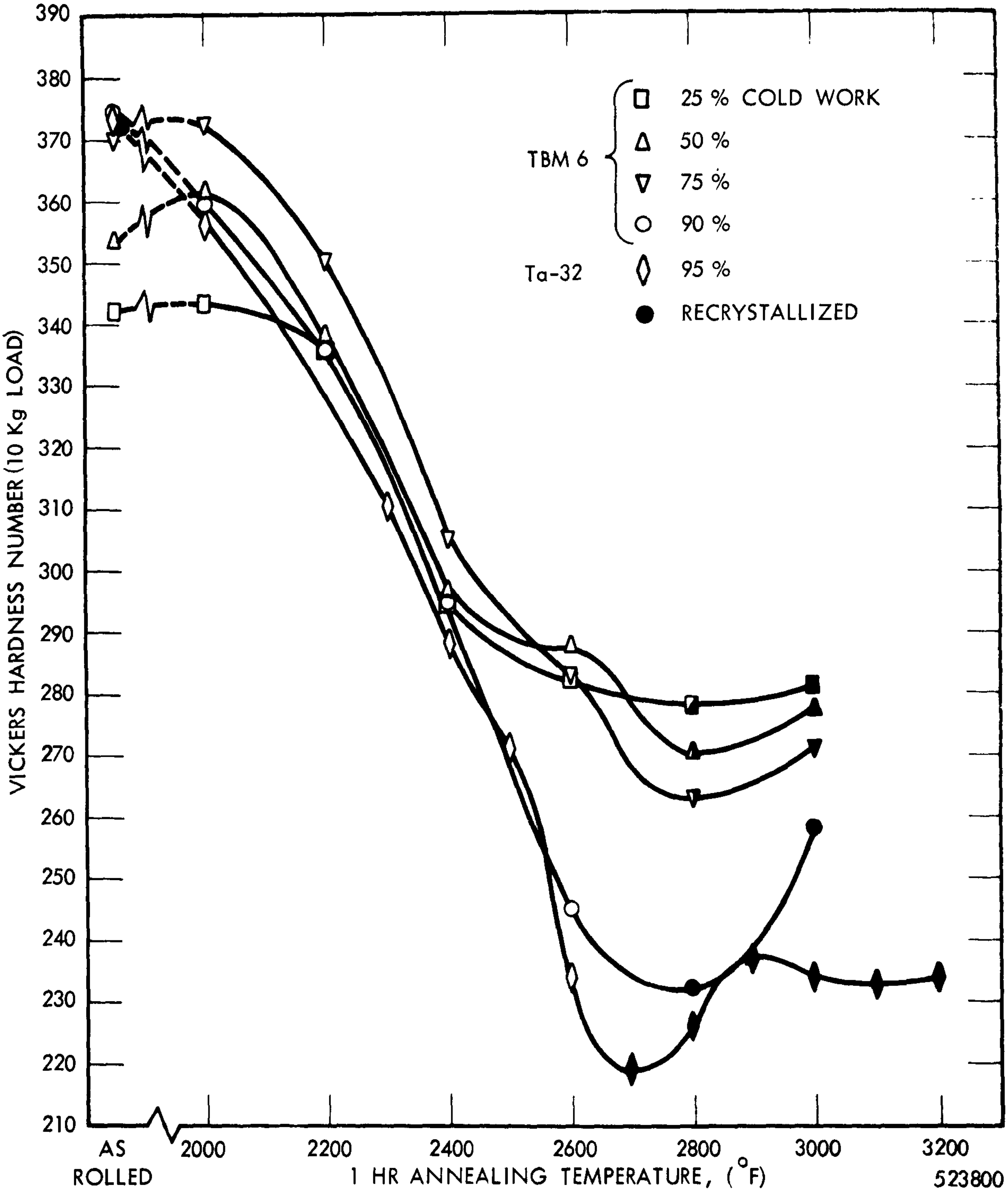


Fig. 23 - The Effect of Annealing Temperature on the Room Temperature Hardness of T-111 Sheet

higher temperatures to initiate and complete the recrystallization process. From the data, it appears that the temperature required to produce a 50% recrystallized structure in a one-hour anneal for T-111 material cold worked 90% or more is approximately 2550°F.

The data also indicated a strong dependency of the hardness of recrystallized material on the amount of cold work received prior to recrystallization. Material cold worked 25% had a hardness of 281 VHN after a recrystallization anneal at 3000°F while material cold worked 95% exhibited the lowest hardness value, 219 VHN, after one hour at 2700°F. This type of behavior has been noted by others. Stewart, Liebermann, and Rowe have shown similar behavior in hardness data for the Cb-1Zr alloy[13]. They suggested a possible interaction between dislocations and a precipitate, occurring in this alloy. They further suggested that the amount of cold work affected the distribution and location of the precipitating phase. Work at Battelle has also shown a pronounced variation in recrystallized hardness with the amount of prior work in a Ta-10Hf-5W alloy, as well as a Ta-30Cb-7.5V alloy[14]. Begley and France reported similar results for electron beam melted columbium. Their work indicated that pure materials display this same type of behavior[15].

Another possible explanation for this behavior has been revealed by the work of Pugh and Hibbard on the rolling texture of pure tantalum[16]. Their work showed that the cold-rolled texture of tantalum was similar to those of other body-centered cubic metals. Isochronal anneals of heavily worked tantalum sheet followed by pole-figure studies showed that annealing treatments caused a sharpening of the rolling texture during recovery, a strengthening of a second texture during recrystallization, a monopoly of the latter texture by a single orientation after recrystallization, and finally a sharpening of that texture after grain growth. This evidence suggests that properties of recrystallized material could very well have been influenced by the degree of preferred orientation induced by cold work.

As noted earlier in this report material from heat Ta-32 cold rolled 65% prior to recrystallization exhibited significantly higher 2400°F tensile properties than material rolled 95% prior to recrystallization. In light of the work of Stewart, Liebermann and Rowe[13], it appears that the difference in properties is a real effect and suggests an area for further investigation. This behavior adds another variable to the long list which must be considered when comparing data from different sources.

Physical Properties

Thermal Expansion of T-111. In conjunction with a Westinghouse-sponsored program, thermal expansion data for T-111 were obtained. The data are included in this report because of their pertinence to the contract activity. Thermal expansion measurements of recrystallized T-111 were made using the dilatometric technique in vacuum in the temperature range 75 to 4400°F. The specimen, 2.00" x 0.250" diameter, was heated by a tantalum resistance element which was programmed to give the specimen a heating and cooling rate of 6.8° per minute. Expansion of the specimen was transmitted to a linear variable differential transformer by means of a tantalum push rod. The expansion of the specimen was continuously plotted by an X-Y recorder during both the heating and cooling cycles. Temperature was monitored by means of a tungsten/tungsten-rhenium thermocouple and optically by a two-color pyrometer (Shawmeter). Vacuum at all times was maintained at 5×10^{-6} Torr or better. No significant change in specimen dimensions was observed after testing.

The data are shown plotted in Figure 24 along with comparative data for unalloyed tantalum[17]. Table 10 lists the average linear coefficients of thermal expansion for recrystallized T-111. The data show the thermal expansion of T-111 to be slightly lower than that of pure Ta.

TABLE 10 - Average Coefficient of Thermal Expansion for T-111

Temperature $^{\circ}F$	Average Coefficient in/in/$^{\circ}F \times 10^{6}$
80 - 500	3.1
80 - 1000	3.5
80 - 1500	3.9
80 - 2000	3.9
80 - 2500	4.0
80 - 3000	4.2
80 - 3500	4.2
80 - 4000	4.2
80 - 4350	4.3

Electrical Resistivity of T-111. Electrical resistivity measurements were made of recrystallized T-111 wire at liquid nitrogen temperature, -320°F; room temperature, 78°F; boiling water temperature, 212°F; and at elevated temperatures, 1475 and 2160°F. Wire 0.057" diameter was used and measurements were made by means of a conventional Kelvin-Bridge. For the high temperature measurements the sample was heated in vacuum at pressures below 5×10^{-5} Torr. Figure 25 shows the electrical resistance of T-111 as a function of temperature along with comparative data for unalloyed tantalum[17]. As would be expected, the electrical resistivity of T-111 was significantly higher than pure Ta over the temperature range investigated.

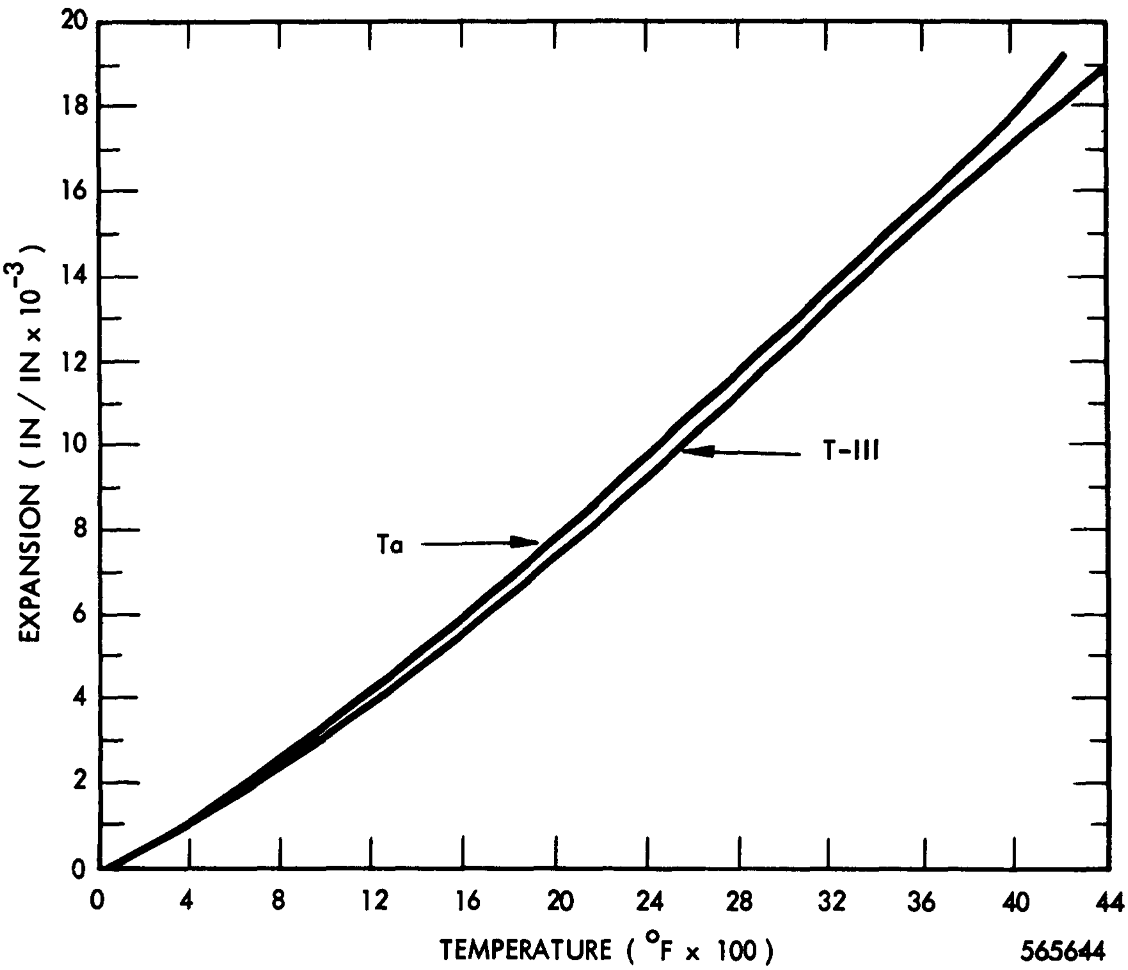


Fig. 24 - Thermal Expansion of Tantalum and T-111

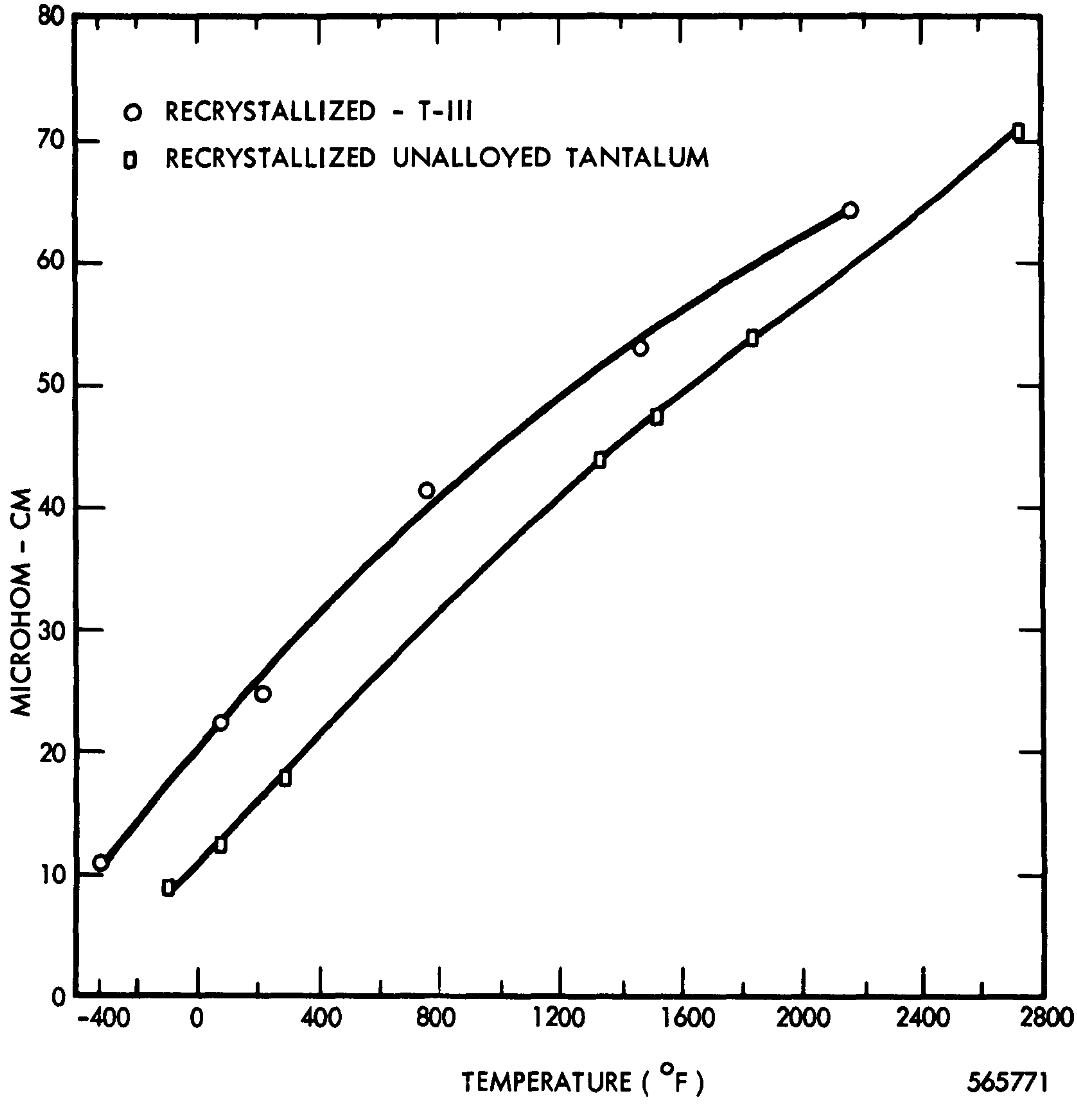


Fig. 25 - Electrical Resistivity of Tantalum and T-111

THE EFFECTS OF INTERSTITIAL ELEMENTS ON THE PROPERTIES OF T-111

Interstitial elements, whether present as impurities or as intentional additions, have pronounced effects on the properties of refractory metals and their alloys. It is, therefore, desirable that their individual and collective effects on a particular alloy be known so that their beneficial aspects can be exploited and their detrimental aspects be avoided. The objective of this phase of the program was to determine the tolerability limits of T-111 for carbon, oxygen, and nitrogen; that is, to determine the maximum concentration of each interstitial element which can be present in T-111 without compromising fabricability, weldability, and mechanical strength.

This objective was achieved by adding to the basic T-111 composition specified amounts of each individual interstitial element while the conditions of processing and evaluation were held constant. Nominal concentration levels of 0.02 and 0.05 w/o for individual elements were used. One alloy which contained 0.01 w/o of each of the elements carbon, oxygen, and nitrogen was investigated to determine if an interaction between interstitial occurred.

Evaluation was conducted on the basis of fabricability, weldability and mechanical strength. Fabricability was determined using procedures which were successful in producing sheet from as-cast button ingots of T-111. Weldability was judged using weld bend transition temperature as the criterion. Mechanical properties were determined at -320°, 75°, 2400°, and 3000°F and compared to low interstitial T-111. Test specimen design and test conditions were identical to those used to evaluate low interstitial T-111.

Experimental Procedure

The investigation of the effects of interstitial elements in T-111 was conducted using sheet material made from button ingots. The ingots were prepared and processed to sheet as previously described in this report. Carbon, oxygen, and nitrogen were added in the form of master alloys. The interstitial analyses of the as-cast ingots are given in Table 11.

The button ingots were aluminide coated, forged to approximately 50% reduction at 2200°F, conditioned, recrystallized one hour at 3000°F, and cold rolled to 0.055" thick sheet. All alloys forged without difficulty except TBM-17 and 18. These button heats contained approximately 0.07 w/o of oxygen and nitrogen respectively. During forging of these particular alloys peripheral cracking occurred, with the severity of the cracking being more pronounced in the nitrogen-containing alloy. Sufficient sound material was obtained from all heats to enable evaluation of the rolling process after recrystallization. All heats were successfully cold rolled to 0.055" sheet. Sufficient sheet was produced of each heat except TBM-18 to enable evaluation of weld characteristics and mechanical properties. The low yield of sheet material from heat TBM-18 limited the investigation of mechanical properties and prevented the evaluation of the weld properties of this alloy.

TABLE 11 - Chemical Analyses of Interstitial-Containing T-111 Alloy Button Heats*

Heat No.	Nominal Composition T-111 plus	Chemical Analysis (w/o)		
		C	O	N
TBM-10	0.02 C	0.0215	0.0011	0.0016
TBM-11	0.02 O	0.0042	0.0127	0.0045
TBM-12	0.02 N	0.0034	0.0018	0.0174
TBM-15	0.01 C + 0.01 O + 0.01 N	0.0084	0.0131	0.0111
TBM-16	0.05 C	0.0520	0.0009	0.0030
TBM-17	0.05 O	0.0030	0.0700	0.0125
TBM-18	0.05 N	0.0048	0.0024	0.0770
Ta-34	Low Interstitial T-111	0.0011	0.0009	0.0004

*Heats were not analyzed for W and Hf since previous experience has shown that nominal solute composition can be achieved consistently without difficulty.

Mechanical Properties of Interstitial Containing T-111 Alloys

The effects of interstitial additions on the mechanical properties of T-111 were evaluated by means of tensile tests in the temperature range -320 to 3000°F, and by stress-rupture tests at 2400°F.

Tensile Data. Tensile data for T-111 alloys with intentional interstitial additions were obtained at -320, 75, 2400 and 3000°F are listed in Table 12. Included in the table are data for low interstitial T-111 sheet, heat Ta-34. This sheet material was produced from a 3" diameter ingot. Interstitial analysis of this ingot is also included in Table 11. Neglecting the starting ingot sizes and resulting processing variables, which undoubtedly affects the tensile properties of these alloys to some degree, the test results are quite revealing and show the pronounced effect that interstitial elements have on the properties of T-111. Tensile data for T-111 of varying interstitial levels are summarized in Figure 26.

The carbon containing alloys of T-111 demonstrated a significant increase in both yield and ultimate strength over values for low interstitial material at all the temperatures tested. T-111 plus 0.05 carbon appeared to offer only slight, if any, improvement over the alloy containing 0.02 w/o carbon. Elongation values at low temperature varied slightly with carbon content. At elevated temperatures elongation increased with a diminishing effect as the carbon level was raised.

TABLE 12 - Low and High Temperature Tensile Properties of Interstitial-Containing T-111 Alloys

Heat No.	Temperature (^{o}F)	Ultimate Tensile Strength (ksi)	Yield Strength 0.2% Offset (ksi)	Elongation (%)	Remarks
TBM-10 T-111 + 0.02C	-320 75 2400 3000	171.2 108.7 53.7 20.4	153.8 89.75 35.7 19.7	23 26 34 78	Material reduced 70% Recrystallized 1 hr. at 3000^{o}F
TBM-16 T-111 + 0.05C	-320 75 2400 3000	170.4 108.7 54.0 21.4	148.6 82.4 35.3 21.4	26 29 23 68	Material reduced 68% Recrystallized 1 hr. at 3000^{o}F
TBM-11 T-111 + 0.02O	-320 75 2400 3000	148.25 88.8 41.7 15.6	137.8 78.7 25.8 14.9	33 32 38 52	Material reduced 70% Recrystallized 1 hr. at 3000^{o}F
TBM-17 T-111 + 0.05O	-320 75 2400 3000	152.7 88.7 39.7 18.0	140.3 78.0 26.4 17.8	34 32 50 58	Material reduced 68% Recrystallized 1 hr. at 3000^{o}F
TBM-12 T-111 + 0.02N	-320 75 2400 3000	187.2 112.2 58.0 18.9	178.3 105.7 41.9 18.4	25 29 14 43	Material reduced 70% Recrystallized 1 hr. at 3000^{o}F

(continued)

TABLE 12 - Low and High Temperature Tensile Properties of Interstitial-Containing T-111 Alloys

(continued)

Heat No.	Temperature (^{o}F)	Ultimate Tensile Strength (ksi)	Yield Strength 0.2% Offset (ksi)	Elongation (%)	Remarks
TBM-18	-320	224.9	225.2	18	Material reduced 75%
T-111 + 0.05N	75	133.8	132.3	20	Recrystallized 1 hr. at
	2400	56.5	36.3	24	3000^{o}F
	3000	-	-	-	
TBM-15	-320	188.8	186.0	24	Material reduced 70%
T-111 + 0.01C +	75	117.3	115.0	25	Recrystallized 1 hr. at
0.01O +	2400	55.0	37.9	45	3000^{o}F
0.01N	3000	19.7	19.5	122	
Low Interstitial	-320	147.5	131.0	31	Material reduced 80%
T-111 Ta-34	75	84.0	68.6	31	Recrystallized 1 hr. at
	2400	38.0	26.4	29	3000^{o}F
	3000	15.6	14.5	31	

Strain Rate 0.005 in/in/min at low temperatures
Strain Rate 0.05 in/in/min at high temperatures

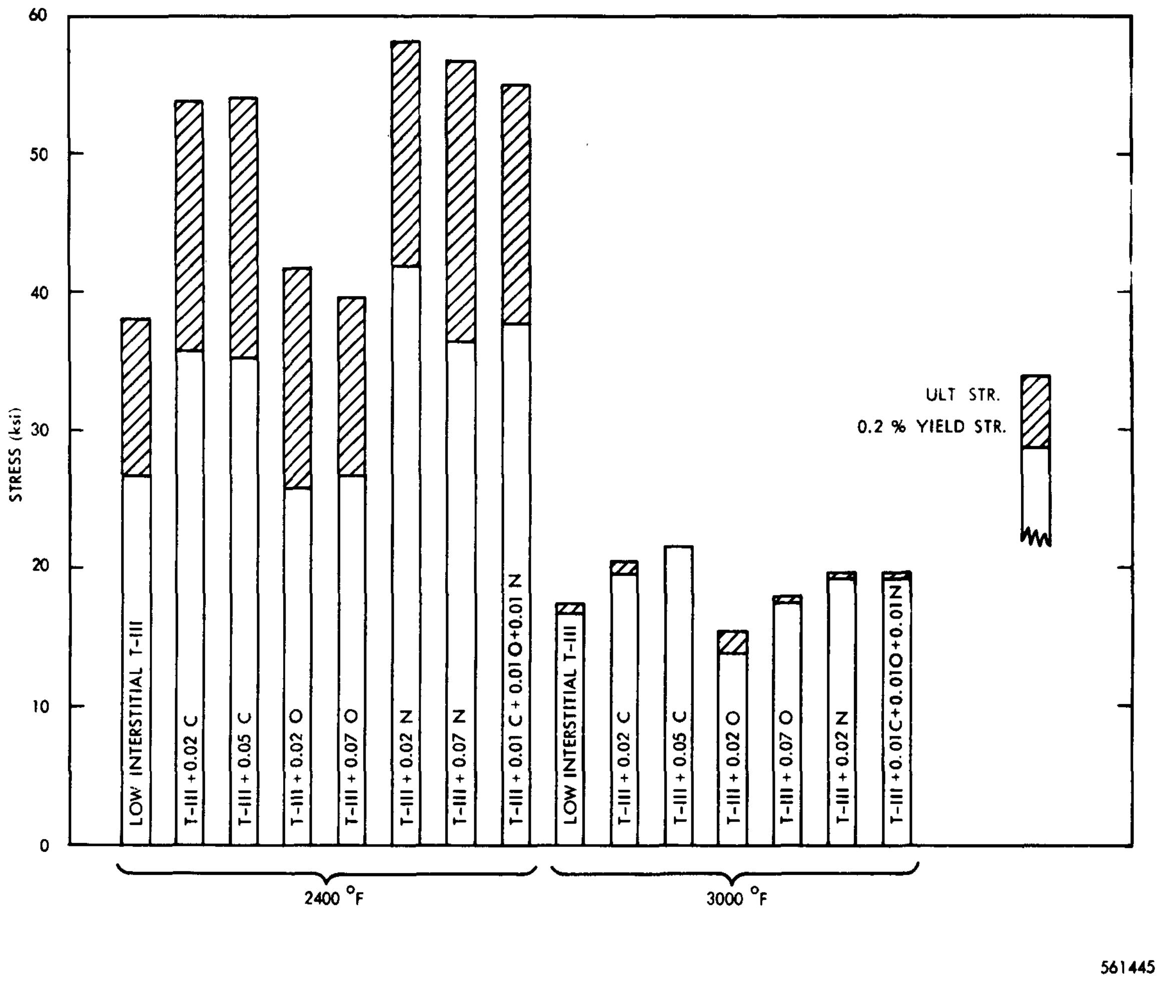


Fig. 26 - Tensile Properties of Interstitial-Containing T-111 Alloys

Oxygen had the least effect on the properties of T-111, showing only a slight increase in tensile properties at all temperatures for both oxygen levels. Elongation values were comparable to the low interstitial T-111 at low temperatures, but became greater as the test temperature was raised.

Nitrogen showed the most pronounced effect at low temperatures with substantial increases in yield and ultimate strengths for both nitrogen levels. The tensile properties of T-111 with 0.02 w/o nitrogen were the highest values achieved in this study. Raising the level of nitrogen to 0.07 did not appear to increase tensile properties over those for the material containing 0.02 w/o nitrogen. Ductility in the nitrogen-containing T-111 at high temperatures was the lowest of all the interstitial-containing alloys.

The alloy with the multiple interstitial additions displayed equally impressive properties at all temperatures. This alloy which contained 0.01 w/o of carbon, oxygen and nitrogen had properties comparable to those which contained 0.02 w/o carbon or nitrogen. While oxygen appears not to be beneficial, the effects of nitrogen and carbon appear to be additive. The ductility of this alloy at 3000°F was extremely high, while comparable to the other alloys at the lower temperatures.

The tensile data for the interstitial-containing alloys show that carbon and nitrogen additions have a pronounced effect on the strength of the base T-111 composition. It is apparent that the increase in high temperature strength is the result of an interstitial-substitutional solute interaction, since interstitial elements per se would not exert any significant strengthening. It should be pointed out that the processing and thermal treatments for the interstitial containing alloys were selected to conform with those used for low interstitial Ta-W-Hf alloys. Variations in prior thermal and mechanical history should significantly affect mechanical properties in interstitial containing T-111 by altering the morphology of dispersed phases.

Stress Rupture Data. The stress rupture properties of interstitial-containing T-111 alloys at 2400°F are given in Tables 13 and 14, and are shown in Figure 27. The one and ten hour rupture properties* demonstrated the significant effects of carbon and nitrogen additions to the basic T-111 composition. Oxygen additions appear to be ineffective, at least under the conditions investigated.

The addition of 0.02 w/o carbon to T-111 raised the one hour rupture stress by 10,000 psi over low interstitial material. For ten-hour life the stress was still 25% higher. Increasing the carbon content to 0.05 w/o raised the stress for one hour rupture life to 46,000 spi and the ten-hour rupture stress to 35,500 psi. This significant increase in stress values was undoubtedly associated with the precipitation of a carbide during the course of the test. The same type of mechanism was probably responsible for the increase in rupture stress for the nitrogen containing alloy. The T-111 + 0.02 w/o nitrogen alloy had comparable properties to the 0.02 w/o carbon containing alloy. Oxygen additions on the other hand showed little or no effect at the 0.02 w/o level and an actual decrease for the 0.07 w/o level. Apparently the solubility relationship between the oxygen in solution in the matrix and

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*The values are approximations only, since they are based on data from only two tests.

oxygen present as an oxide phase was not conducive to precipitation during the 2400°F test. The multi-interstitial containing alloy of T-111 displayed stress rupture properties which were intermediate between the low interstitial T-111 and the material which contained 0.02 w/o of either carbon or nitrogen. At first hand these results indicate that the effects of carbon and nitrogen which totaled 0.02 w/o are not additive. However, the presence of oxygen may actually have been detrimental.

It appears from the results of these tests which are only preliminary in nature, that oxygen levels should be maintained as low as possible, while some addition of carbon and nitrogen are desirable as far as stress rupture properties are concerned.

TABLE 13 - 2400°F Stress Rupture Properties of Interstitial-Containing T-111 Alloys*

Heat No.	Stress (ksi)	Minimum Creep Rate (%/hr)	Transition Time (hrs)	Rupture Time (hrs)	Elongation (%)
TBM-10	40	2.21	3.4	2.1	22
T-111 + 0.02 C	30	0.19	2.7	18.1	27
TBM-16	40	1.38	1.28	3.8	29
T-111 + 0.05 C	30	0.15	6.0	32.3	36
TBM-11	30	3.95	1.4	4.2	38
T-111 + 0.02 O	25	0.50	5.4	39.2	39
TBM-17	30	11.4	1.2	2.1	44
T-111 + 0.07 O	25	1.29	8.1	7.0	14
TBM-12	40	0.97	0.7	2.1	16
T-111 + 0.07 O	30	0.12	4.9	17.7	25
TBM-15	40	2.8	0.4	1.0	24
T-111 + 0.01 C + 0.01 O + 0.01 N	30	0.10	3.7	11.8	29

*All material reduced 70 per cent and recrystallized - one hour at 3000°F.

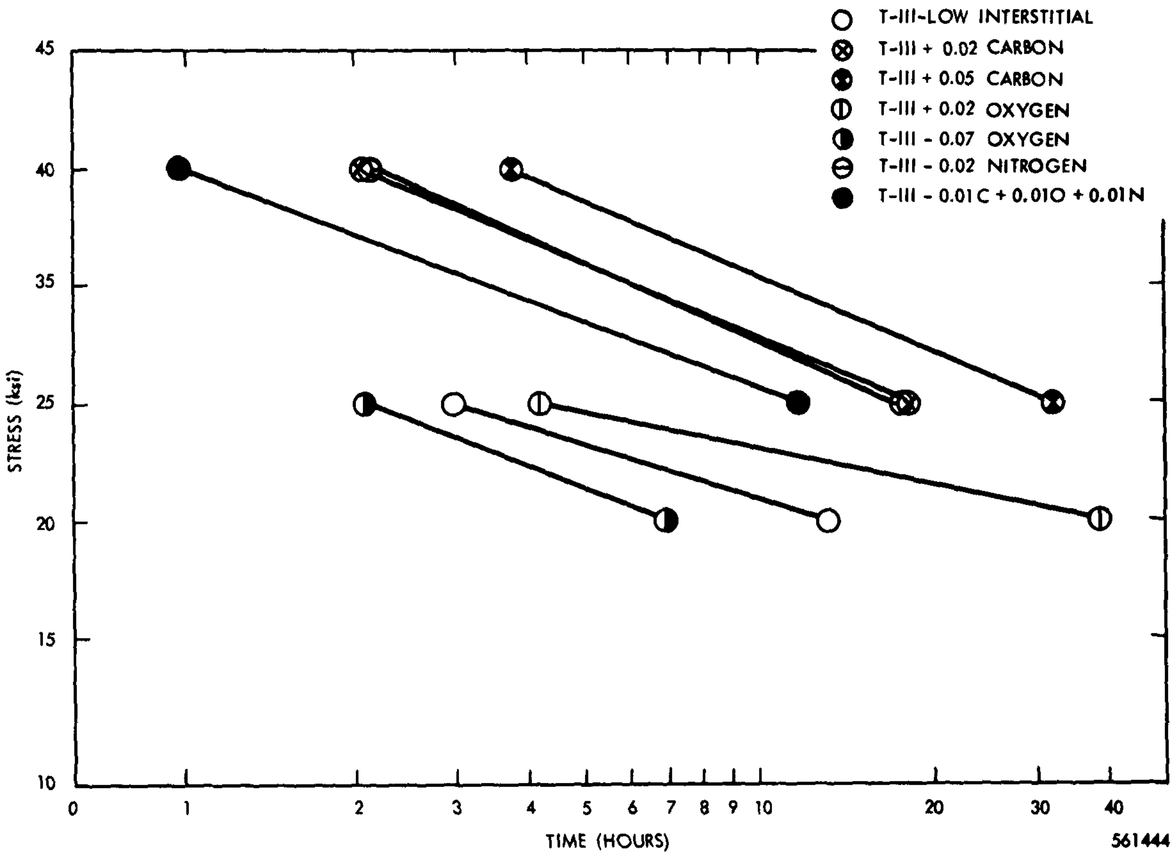


Fig. 27 - 2400°F Stress Rupture Properties Interstitial-Containing T-111 Alloys

TABLE 14 - One and Ten Hour Stress Rupture Properties of Interstitial-Containing T-111 Alloys at 2400°F

Material	Stress for 1 Hr. Rupture Life (ksi)	Stress for 10 Hr. Rupture Life (ksi)
T-111 Low Interstitial	33.5	26.0
T-111 + 0.02 C	43.5	32.5
T-111 + 0.05 C	46.0	35.5
T-111 + 0.02 O	33.0	28.0
T-111 + 0.07 O	33.0	23.5
T-111 + 0.02 N	43.5	32.5
T-111 + 0.01 C + 0.01 O + 0.01 N	40.0	30.5

Weld Characteristics

TIG butt welds of interstitial-containing T-111 sheet material were made in a vacuum purge chamber back-filled with argon using techniques described earlier in this report. The interstitial containing T-111 alloys were stress-relieved one hour at 2000°F prior to welding. All the alloys, which included the low and high individual interstitial-containing alloys as well as the multiple interstitial-containing T-111, were welded without difficulty. No evidence of hot cracking was detected. The low yield of sheet from heat TBM-18 (T-111 + 0.077 w/o nitrogen) prevented weld evaluation of this alloy.

Due to the limited amount of material available and the screening nature of the evaluation, bend specimens 1/4" wide by 1-1/8" long with the weld transverse to the long dimension were used. The specimens were tested at a deflection rate of 1" per minute using a 2T and a 4T mandrel. Specimens were tested in the as-welded condition with no prior surface preparation. Minimum test temperature was limited by equipment to -320°F. Test temperature was varied to determine the transition from ductile to brittle behavior. Specimens which were bent through an angle of 90 degrees or better without visual signs of defects were termed ductile. Those which displayed fissures even though exhibiting considerable yielding in the weld metal and heat-affected zone of the base metal prior to failure were considered brittle.

The results listed in Table 15 show that low concentrations of individual interstitial elements have little effect on weld bend ductility of T-111 at -320°F. Multiple additions totaling 0.03 w/o oxygen plus nitrogen plus carbon also appear to have no detrimental effect on low temperature ductility. Carbon at 0.05 w/o level raised the 2T bend transition to room temperature and the 4T bend transition to -200°F. Oxygen at the higher

TABLE 15 - Weld Bend Ductility of Interstitial-Containing T-111 Alloys

Heat No.	Composition	Bend Transition Temperature	
		2T bend radius	4T bend radius
TBM-10	T-111 + 0.02 C	-320°F	-320°F
TBM-11	T-111 + 0.02 O	-320°F	-320°F
TBM-12	T-111 + 0.02 N	-320°F	-320°F
TBM-15	T-111 + 0.01 C + 0.01 O + 0.01 N	-320°F	-320°F
TBM-16	T-111 + 0.05 C	RT	-200°F
TBM-17	T-111 + 0.04 O	-320°F	-320°F

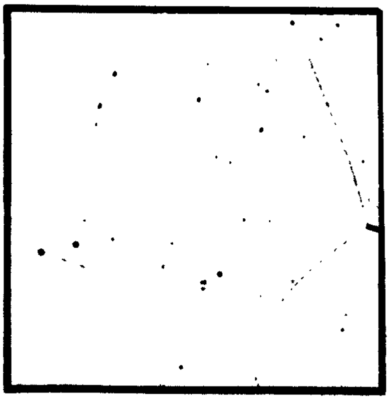
concentration did not affect the bend transition for either 2 or 4T bend conditions. These results indicate a high tolerance for interstitial elements in T-111 with respect to weld bend ductility. These results are encouraging in view of the increased high temperature strength displayed by the interstitial containing alloys.

Aging Studies. The interaction of interstitial elements with Group IV-A elements (Ti, Zr, and Hf) in columbium and tantalum alloys to form carbides, oxides, and nitride phases has been shown to exert a pronounced effect on mechanical properties[18, 21, 22]. To explore possible heat treatment effects, preliminary aging studies were conducted on the T-111 alloy which contained 0.02 w/o of the individual-interstitial additions.

Samples of the as-rolled sheet from heats TBM-10, 11, and 12 were solution heat treated for one hour at an arbitrarily selected temperature of 3500°F and quenched in an attempt to produce a single phase microstructure. Quenching was accomplished by dropping the specimens from the hot zone of a vacuum furnace onto a copper plate which was cooled with liquid nitrogen. The specimens, which were wrapped in Ta foil, cooled to below red heat in 15 seconds or less. Metallographic examination showed the carbon and nitrogen containing alloys to be essentially single phase, as in the photomicrographs of Figures 28 and 29. The oxygen containing alloy showed some evidence of incomplete solution of a second phase (Figure 29b). Samples of the solution treated material were aged in vacuum for one hour at 1500, 1650 and 1800°F and room temperature hardness measurements were then obtained. These data are shown in Table 16.

TABLE 16 - Hardness of Interstitial-Containing T-111 after Various Heat Treatments

Condition	Hardness (VHN)		
	TBM-10 T-111 + 0.02 C	TBM-11 T-111 + 0.02 O	TBM-12 T-111 + 0.02 N
Rolled and Recrystallized	258	230	272
Solution heat treated 1 hr at 3500°F and quenched	279	236	279
Solution heat treated 1 hr at 3500°F and quenched Aged 1 hr at 1500°F	235	231	281
Solution heat treated 1 hr at 3500°F and quenched Aged 1 hr at 1650°F	238	222	289
Solution heat treated 1 hr at 3500°F and quenched Aged 1 hr at 1800°F	238	224	287



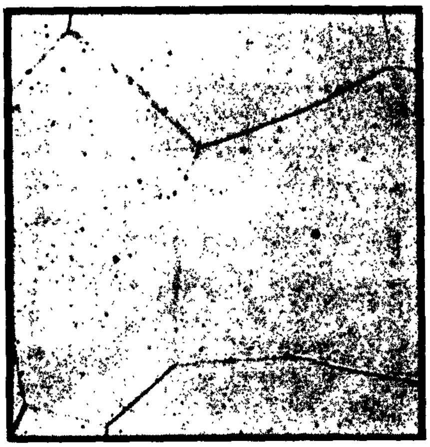
a) T-III + 0.02 % C
SOLUTION HEAT TREATED
1 HR. AT 3500 °F
AND QUENCHED

b) T-III + 0.02 % C
SOLUTION HEAT TREATED
1 HR. AT 3500 °F
QUENCHED AND AGED
1 HR. AT 1650 °F

c) T-III + 0.02 % O
SOLUTION HEAT TREATED
1 HR. AT 3500 °F
AND QUENCHED

Fig. 28 - Microstructures of Carbon and Oxygen-Containing T-111 Alloys after Various Heat Treatments

a) T-III + 0.02 % N
SOLUTION HEAT TREATED
1 HR. AT 3500 °F
AND QUENCHED



b) T-III . 0.02 % N
SOLUTION HEAT TREATED
1 HR. AT 3500 °F
QUENCHED AND AGED
1 HR. AT 1850 °F

Fig. 29 - Microstructures of Nitrogen-Containing T-111 after Various Heat Treatments

The hardness values for the rolled and recrystallized conditions were for material which was annealed for one hour at 3000°F and furnace cooled. The hardness of the carbon containing T-111 was increased from 258 to 279 VHN using the solution heat treatment and quenching procedure, while the oxygen and nitrogen containing material exhibited only a slight increase in hardness.

The hardness of the carbon containing material dropped to 235 VHN after one hour at 1500°F. Aging at 1650 and 1800°F caused no additional change in hardness. The microstructure of the aged material (Figure 29c) showed that precipitation of platelets had occurred at the 1500°F temperature. The low hardness values plus the precipitation of an abundant second phase, likely a carbide, indicate that the carbon containing T-111 alloy ages at temperatures below 1500°F.

The oxygen containing T-111 showed little response to solution heat treatment and aging treatment. Solution temperature may not have been high enough or solution time at temperature long enough to allow complete dissolution of HfO_2. Begley, et al, have noted in columbium alloys containing hafnium that the addition of oxygen caused a reduction in the hardness of the material[18]. As the oxygen concentration was raised, the hardness values continued to decrease proportionally. This reduction in hardness was attributed to the removal of solute hafnium by the formation of HfO_2 which apparently contributes little or no strength to the matrix. This same explanation appears applicable in the case of tantalum base alloys. The achievement of classical aging phenomenon in tantalum base alloys containing hafnium and oxygen may require higher solution treating temperatures to achieve complete dissociation and solution of the hafnium and oxygen in the matrix. The nitrogen containing T-111, however, displayed encouraging results. The solution treated and quenched material exhibited a clean single phase microstructure and a hardness of 279 VHN. On aging the hardness values increased sufficiently to indicate that classical aging response may have occurred.

Although this study was quite limited some pertinent facts can be drawn from the data. For the given conditions it appears that T-111 containing 0.02 w/o carbon can be solution treated at 3500°F, but that overaging occurs at a temperature of 1500°F in a period of one hour. In the case of oxygen, classical aging phenomena were not observed under the particular conditions investigated. Nitrogen additions showed promise of significant heat treatment response. These results indicate that it is possible to augment the high temperature properties of T-111 by disperse phase strengthening without adversely affecting the excellent fabricability and weldability inherent in the T-111 alloy. More detailed studies of heat treatment effects of interstitial containing Ta-W-Hf alloys are being carried out in an extension of this program.

Recovery and Recrystallization Behavior

The sheet material which was produced from interstitial-containing T-111 button ingots was evaluated to determine recrystallization behavior. Hardness measurements were taken on forged and recrystallized slabs prior to final cold rolling to sheet. The hardness values of Figure 30 show the pronounced effect of nitrogen and carbon, and the seemingly inocuous effect of oxygen, on the hardness of T-111. The T-111 + 0.07 w/o oxygen alloy also contained 0.0125 w/o nitrogen which undoubtedly contributed to the hardness and thus masked the effect of oxygen in this particular alloy. After recrystallization of the forged slabs, the alloys were cold rolled 70% to sheet, and samples were subsequently given a one hour anneal at temperatures ranging from 2000 to 3000°F. The room temperature hardness of the alloys are shown as a function of annealing temperature in Figure 31. Included are reference data from Figure 23 for low interstitial T-111, heat Ta-32, which was reduced 95% prior to annealing.

The data of Figure 31 show that the oxygen containing T-111 alloys exhibit the normal decrease in hardness with increased annealing temperature, with essentially complete hardness recovery occurring after annealing 1 hour at 2600°F. As noted in a previous section of this report, a stable HfO_2 phase is apparently formed in the oxygen containing T-111, which is ineffective with regard to hardening the matrix, and results only in reducing the concentration of hafnium in solution in the matrix. The reduction of hafnium in solution is reflected in the lower hardness of the alloy. The presence of 0.0125 w/o nitrogen in the T-111 + 0.07 oxygen alloy accounts for somewhat higher hardness levels than ordinarily would be expected. Begley, et al, found in Cb-Hf alloys that hardness values decreased as oxygen level increased when other interstitial elements were maintained at low levels.[18] Metallographic examination indicated the 0.02 w/o oxygen alloy to be completely recrystallized after annealing 1 hour at 2600°F, and the 0.07 oxygen alloy after 1 hour at 2400°F.

Carbon and nitrogen additions in T-111 result in somewhat different behavior than the oxygen containing alloys. While all the alloys show the general decrease in hardness with increasing annealing temperature, the 0.05 w/o C alloy and the alloy containing multiple interstitial additions show perturbations in the hardness-annealing temperature curve at 2600°F indicative of precipitation hardening effects. Several of the alloys also exhibit hardness increases at annealing temperatures of 2800 and 3000°F.

In the temperature range 2000 to 2600°F general precipitation occurs in the carbon and nitrogen containing alloys, the extent of precipitation increasing with increasing temperature. In the range 2500 to 2700°F (the recrystallization range of low interstitial T-111) the precipitates inhibit grain growth and generally obscure the recrystallization process, making metallographic determination of recrystallization difficult. However, there appears to be no pronounced effect of carbon and nitrogen additions in the recrystallization range.

While detailed studies of precipitation phenomena were not carried out, the hardness data indicate that these alloys show rather pronounced response to heat treatment. The results of the recrystallization study illustrate an area of investigation which possesses potential for exploitation. The precipitation reaction occurring in these alloys merits further investigation.

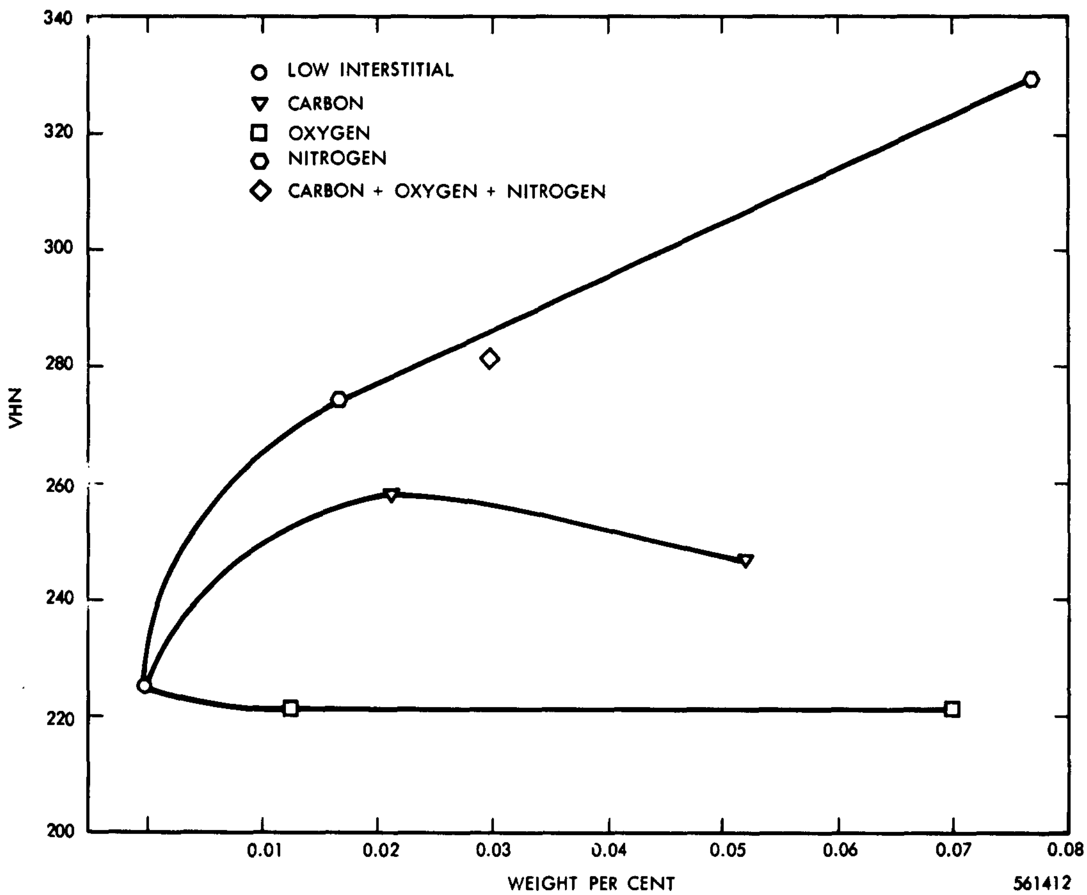


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Fig. 30 - The Effect of Interstitial Additions on the Recrystallized Hardness of T-111 Alloys

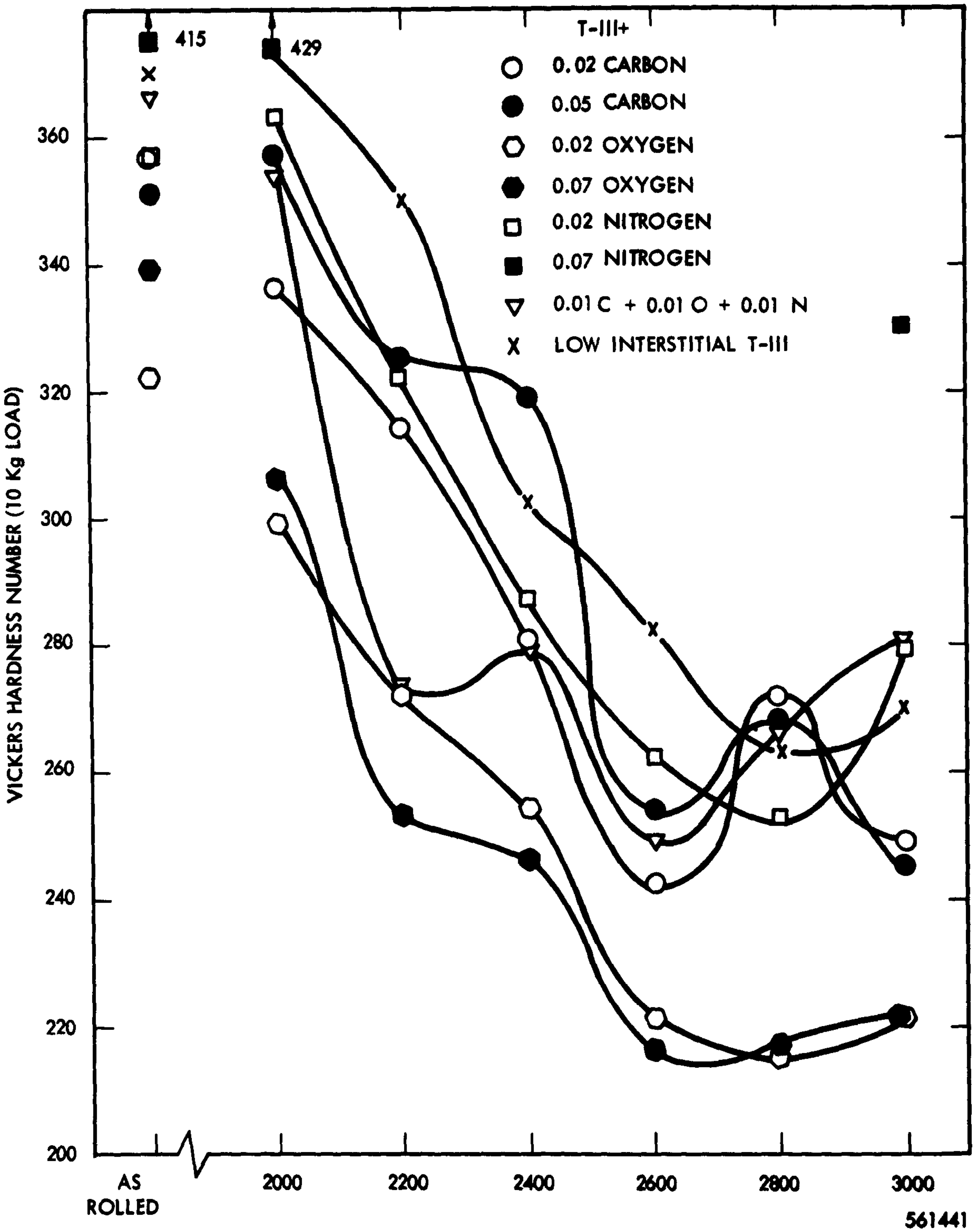

Fig. 31 - The Effect of Annealing Temperature on the Room Temperature Hardness of Interstitial-Containing T-111 Alloys

Summary

Individual interstitial additions to T-111 on the order of 0.02 w/o do not adversely affect workability or weldability. However, a substantial increase in mechanical properties is achieved by the addition of 0.02 w/o carbon and/or nitrogen. Oxygen additions appear to have little or no effect on properties. When the carbon content of T-111 is raised to 0.05 w/o, an increase in ductile to brittle weld bend transition temperature is observed. High nitrogen, 0.07 w/o adversely affects the workability of T-111. In general, interstitials in the range to 0.03 w/o can be tolerated without adversely affecting workability or weldability. The beneficial effects of carbon and nitrogen on mechanical properties make intentional additions in the amount of at least 0.03 w/o desirable.

OPTIMIZATION OF TUNGSTEN AND HAFNIUM IN T-111

The excellent fabricability and weldability displayed by T-111 indicated the possibility of increasing the solute concentrations to higher levels to obtain increased elevated temperature properties without undue sacrifice of the excellent ductility of tantalum. This approach represented a departure from the customary procedure used in refractory metal alloy development of first investigating high temperature strength and then evaluating joinability and fabricability. This procedure has lead to many difficulties in the past. To minimize this problem, weldability and fabricability were used as major evaluating criteria along with elevated temperature properties.

Experimental Procedure

In the evaluation of the effect of higher solute concentrations on the properties of T-111, alloys which contained a total solute content of 14 w/o were prepared with varying W/Hf ratios. One alloy which contained 18 w/o solute addition was also included. The investigation was conducted using sheet material which was prepared in accordance with procedures outlined earlier in this report.

The alloys which were prepared are listed in Table 17, along with chemical analyses. Interstitial elements were maintained at low levels in all heats. It was noted during the melting operation that the heats TBM-2 and TBM-5, both of which contained 6 w/o Hf, cracked during solidification. Sufficient sound material was obtained from these heats to allow continued evaluation. However, the alloy TBM-5, Ta-12W-6Hf, cracked severely during the forging operation, preventing further evaluation of this composition. The remaining four heats were successfully converted to sheet. These alloys were evaluated for high and low temperature tensile properties, recrystallization behavior and weldability.

Mechanical Properties of Ta-W-Hf Alloys

The tensile properties of the tantalum alloy containing 14 w/o total solute concentration are given in Table 18.

Tensile data were obtained for Ta-W-Hf alloys at -320, RT, 2400 and 3000°F. Stress rupture tests were also conducted at 2400°F. All samples were recrystallized by annealing 1 hour at 3000°F prior to testing.

Tensile Data. Tensile properties of alloys having varying W/Hf ratios (14 w/o total solute addition) are listed in Table 18. The increase in solute concentration resulted in significantly higher tensile properties with respect to T-111 over the entire temperature range. All of the alloys exhibited ultimate tensile strength values in excess of 20,000 psi at 3000°F. The effect of tungsten-hafnium ratio on the elevated temperature properties of Ta-W-Hf alloys is illustrated in Figure 32. The data for heat TBM-14 (Ta-11.2W-2.8Hf) are somewhat inconsistent with respect to the other alloys at 2400 and 3000°F. Examination of the tensile specimens indicated the presence of tungsten segregation, which accounted for the anomalous results.

TABLE 17 - Chemical Analyses of Ta-W-Hf Alloy Heats

Heat No.	Nominal Composition (w/o)		Chemical Analyses (w/o)				
	W	Hf	W	Hf	O	N	C
TBM-2	8	6	8.06	6.15	0.0014	0.0016	0.0018
TBM-3	10	4	10.15	4.06	0.0018	0.0014	0.0026
TBM-4	12	2	11.80	2.18	0.0015	0.0011	0.0022
TBM-5	12	6	12.09	6.09	0.0013	0.0006	0.0020
TBM-14	8	2	-	-	0.0019	0.0074	0.0034

TABLE 18 - Tensile Properties of Ta-W-Hf Alloys

Heat	Temperature ($^{\circ}$F)	Ultimate Tensile Strength (ksi)	Yield Strength 0.2% Offset (ksi)	Elongation (%)	Remarks
TBM-2 Ta-8W-6Hf	-320	172.0	157.0	25	Material reduced 80% Recrystallized 1 hr. at 3000°F
	75	106.8	96.2	32	
	2400	52.3	39.7	23	
	3000	20.0	18.6	81	
TBM-3 Ta-10W-4Hf	-320	172.0	163.8	23	Material reduced 80% Recrystallized 1 hr. at 3000°F
	75	106.0	97.2	26	
	2400	49.6	37.0	32	
	3000	25.8	25.3	67	
TBM-14 Ta-11.2W-2.8Hf	-320	-	-	-	Material reduced 70% Recrystallized 1 hr. at 3000°F
	75	104.3	97.6	14*	
	2400	42.2	29.3	31	
	3000	20.1	20.1	38	
TBM-4 Ta-12W-2Hf	-320	172.5	159.0	29	Material reduced 80% Recrystallized 1 hr. at 3000°F
	75	105.4	96.8	32	
	2400	56.3	40.0	36	
	3000	24.2	23.3	57	

Strain Rate 0.005 in/in/min at low temperatures
Strain Rate 0.05 in/in/min at high temperatures

*Equipment malfunctioned. Test stopped after ultimate was reached.

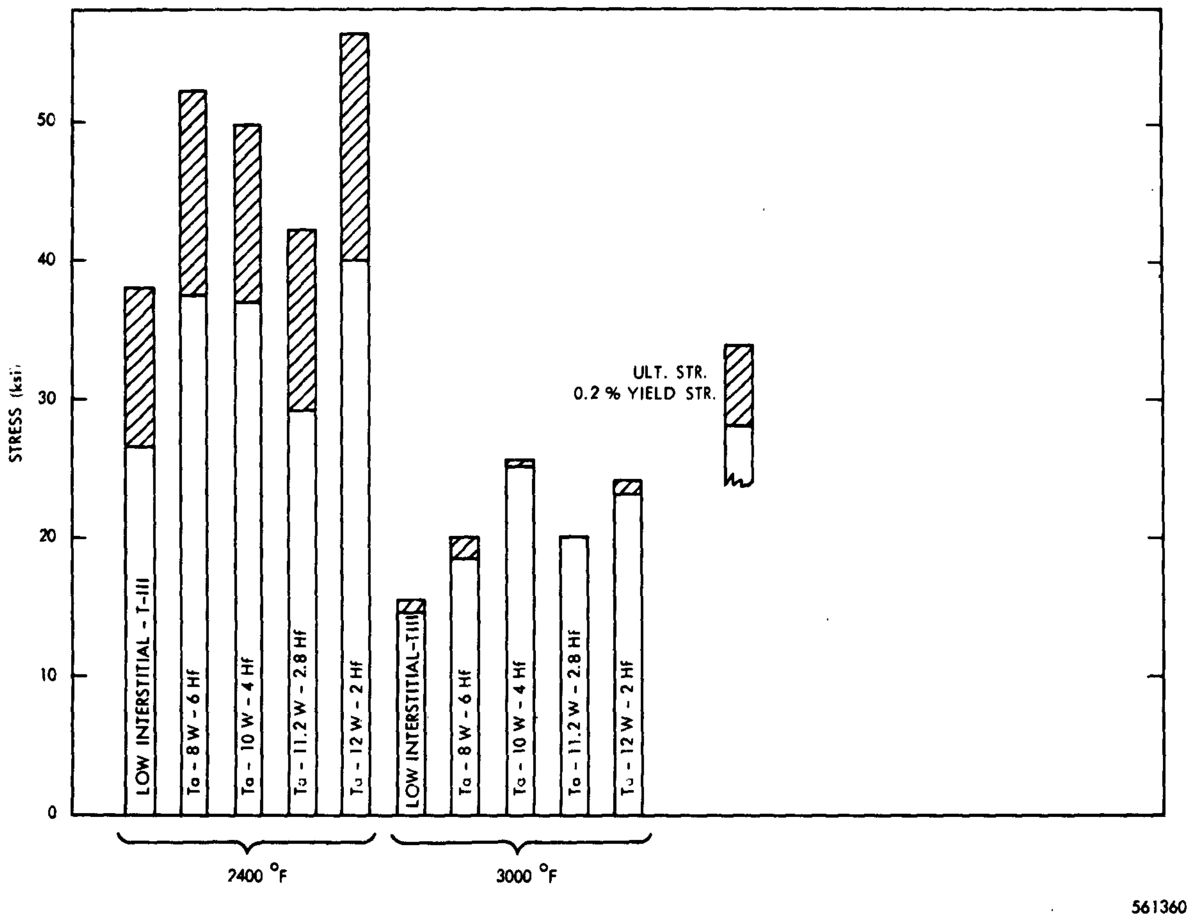

Fig. 32 - Tensile Properties of Ta-W-Hf Alloys

The strength increase resulting from the higher solute level was achieved without sacrifice in low temperature ductility, as shown by the uniformly high elongation values of the tensile specimens tested at -320°F (Table 18).

Stress Rupture Data. Stress rupture tests of Ta-W-Hf alloys were conducted at 2400°F on the sheet material produced from button ingots. Tests were conducted with the specimens wrapped in Ta foil.

The stress rupture properties of Ta-W-Hf alloys including comparative data for low interstitial T-111 are shown in Figure 33 and listed in Table 19. One and ten hour rupture stress values obtained from these data are shown in Table 20. It must be noted that these results are derived from single point data, hence the data are primarily indicative of a trend of behavior. The trend toward higher stress levels with increasing tungsten content is evident. Tungsten has also been shown to be the most effective solution strengthener for elevated temperature creep strength in columbium[22].

Weld Characteristics

The Ta-W-Hf alloy sheets produced from the button heats were stress-relieved for one hour at 2000°F and then butt welded in the same manner as the T-111 baseline material previously described in this report. A photograph of the as-welded sheet is shown in Figure 34.

Due to the limited amount of material available and the qualitative nature of the evaluation, bend specimens 1/4" wide by 1-1/8" long with the weld running transverse to the long dimensions were used. The specimens were tested at a deflection rate of 1" per minute using a 2T and a 4T mandrel. Test temperature was varied to determine the transition from ductile behavior. Specimens which were bent through an angle of 90 degrees or better without visual signs of defects were termed ductile. Figure 35 shows the transition temperature for the 2 and 4T bend radii as a function of tungsten-hafnium ratio for the alloys. The data indicate a trend toward a ductility maximum at a tungsten-hafnium ratio of 4 to 1. No obvious explanation for this behavior is available at the present time. The tungsten-hafnium ratio for the T-111 alloy is 4 to 1 and the transition temperature below -320°F. The comparison is not truly valid since all the alloys evaluated contain 14 w/o total alloy content, except the T-111 alloy which has only a total of 10 w/o alloy addition. However, the ductility of T-111, which has a weld bend transition well below -320°F, gives strong supporting evidence to the existence of the bend transition minimum, as shown by the alloys containing 14 w/o solute concentration. The addition of 4 w/o solute to the T-111 composition apparently raised the bend transition temperature for the 2T condition to -200°F. Increasing the solute concentration to higher levels would probably raise the transition temperature to room temperature or above.

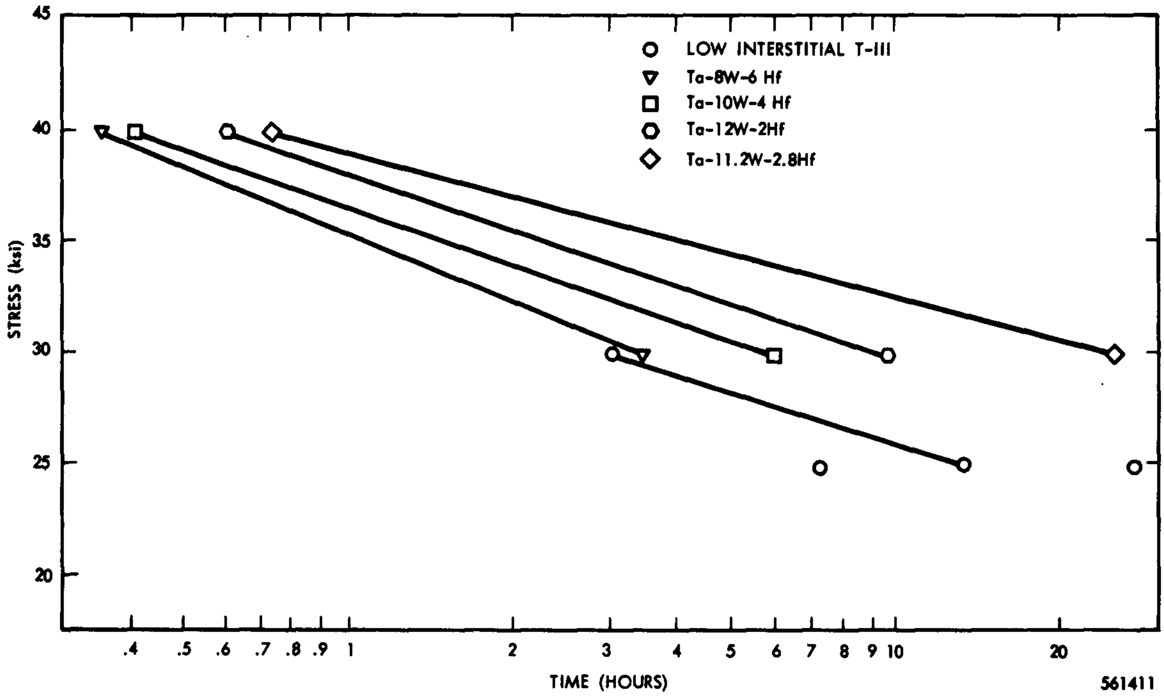

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Fig. 33 - 2400°F Stress Rupture Properties of Ta-W-Hf Alloys

TABLE 19 - 2400°F Stress Rupture Properties of Ta-W-Hf Alloys

Heat No.	Stress (ksi)	Rupture Time (hrs)	Elongation (%)	Transition Time (hrs)	Minimum Creep Rate	Remarks
TBM-2 Ta-8W-6Hf	40 30	0.35 3.4	16 45	0.3 1.3	25.0 5.1	Material reduced 80% Recrystallized 1 hr/3000°F
TBM-3 Ta-10W-4Hf	40 30	0.40 6.0	26 33	0.2 2.5	32.5 2.9	Material reduced 80% Recrystallized 1 hr/3000°F
TBM-4 Ta-12W-2Hf	40 30	0.60 9.5	30 32	0.3 5.0	38.0 2.2	Material reduced 80% Recrystallized 1 hr/3000°F
TBM-14 Ta-11.2W-2.8 Hf	40 30	0.72 25.0	32 32	10.3 3.8	18.2 0.3	Material reduced 70% Recrystallized 1 hr/3000°F

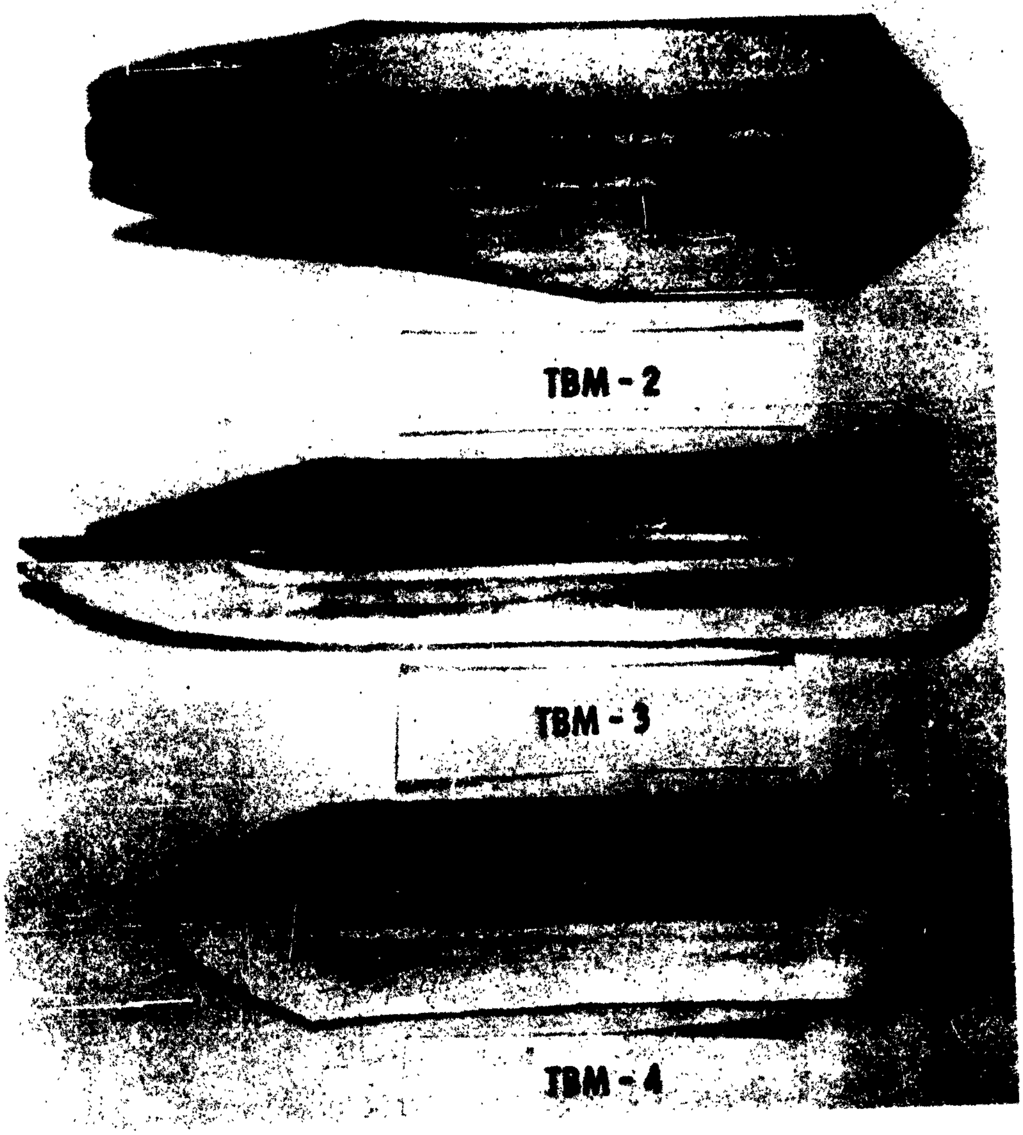

Fig. 34 - As Welded Ta-W-Hf Alloy Sheets

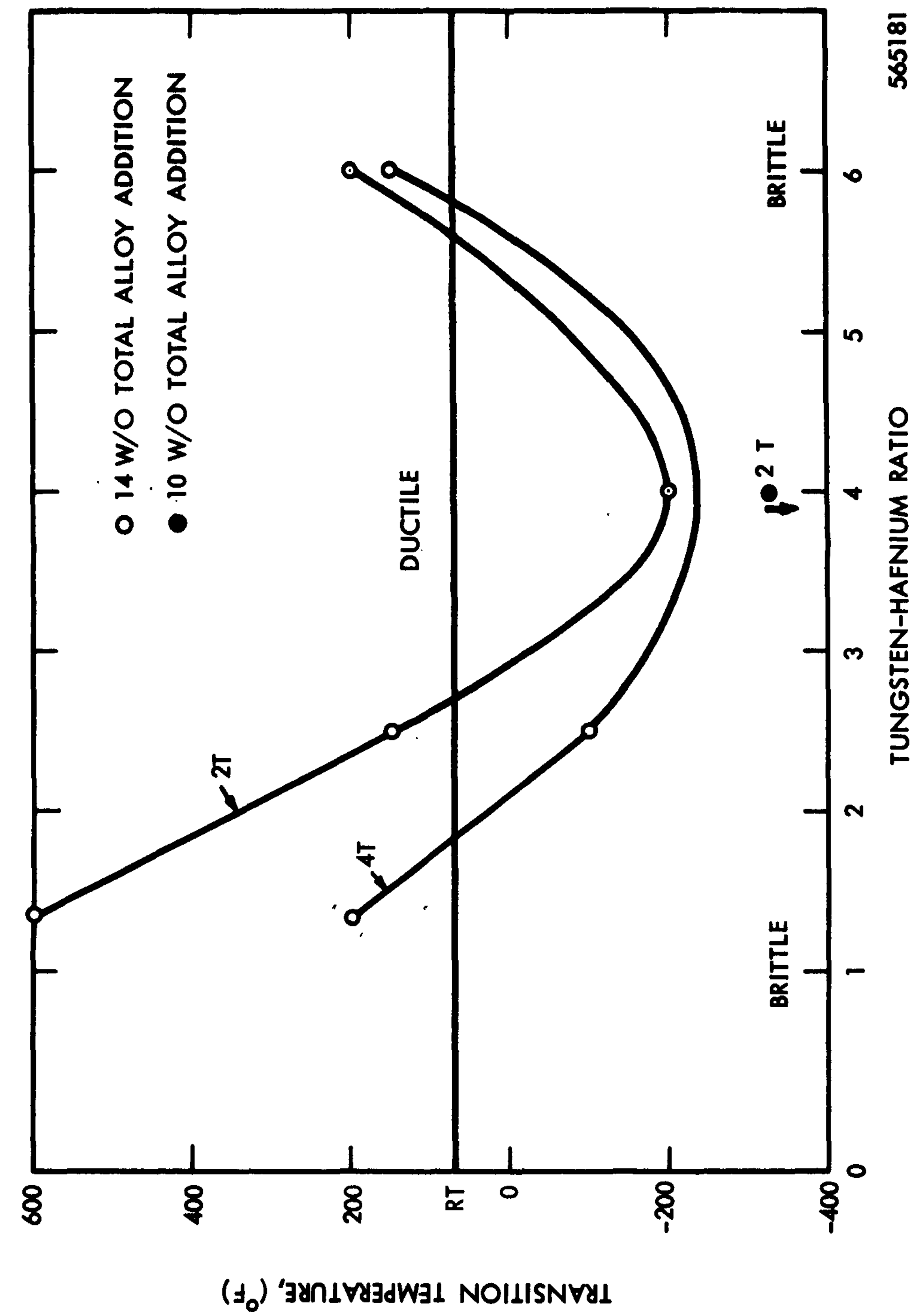

Fig. 35 - Weld Bend Ductility of Ta-W-Hf Alloy Sheet

TABLE 20 - One and Ten Hour Stress Rupture Properties of Ta-W-Hf Alloys at 2400°F

Heat No.	Stress for 1 hour life (ksi)	Stress for 10 hour life (ksi)
TBM-2 Ta-8W-6Hf	35.3	25.3
TBM-3 Ta-10W-4Hf	36.5	28.0
TBM-4 Ta-12W-2Hf	38.0	29.5
TBM-14 Ta-11.2W-2.8Hf	39.0	32.5
Ta-34 Ta-8W-2Hf	33.5	26.0

Recrystallization Behavior

Samples of Ta-W-Hf alloys containing 14 w/o total solute concentration were annealed one hour at temperatures ranging from 2000 to 3000°F to evaluate recrystallization behavior. All material was cold rolled 80% prior to annealing. The extent of recrystallization was established by means of hardness measurements and metallographic examination.

The effect of annealing temperature on the room temperature hardness of the specimens is shown in Figure 36. The data show the normal decrease in hardness with increasing annealing temperature, with some recovery occurring after a one hour anneal at 2000°F. Hardness decreased rapidly in the 2200 to 2600°F range, with minimum hardness occurring after a one hour anneal at 2800°F. Hardness values were generally in order of tungsten content, the alloys of highest tungsten level having the highest hardness. In the fully recrystallized condition, the hardness was independent of tungsten-hafnium ratio.

Metallographic examination showed recrystallization to be initiated after annealing 1 hour at 2400°F for the Ta-8W-6Hf alloy. The alloys of higher tungsten content did not show evidence of recrystallization after a 2600°F anneal. All alloys were fully recrystallized after annealing one hour at 2800°F. The hardness of these alloys in the as-recrystallized condition is 50 VHN higher than most of low interstitial T-111. The increase in hardness is

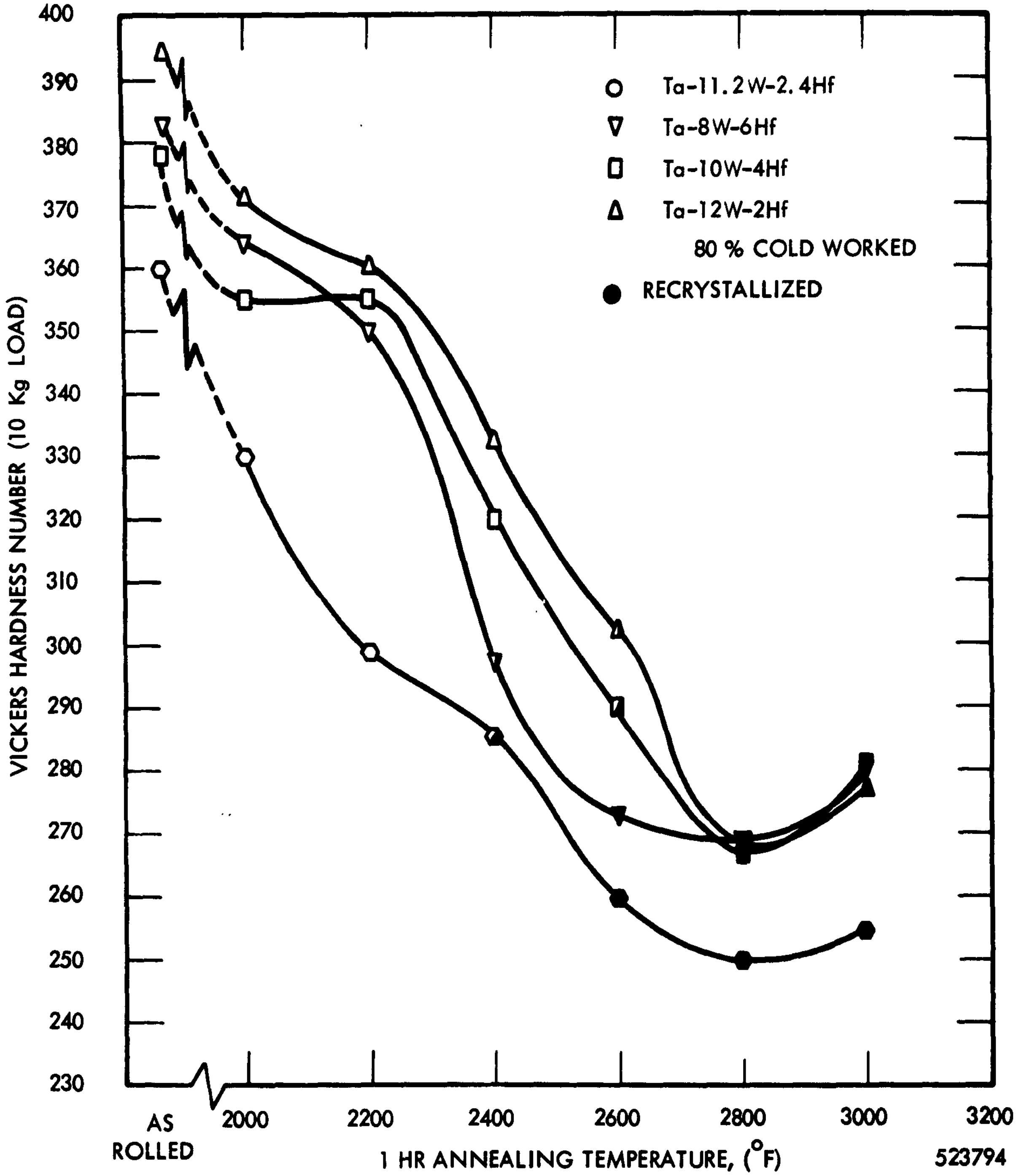

Fig. 36 - The Effect of Annealing Temperature on the Room Temperature Hardness of Ta-W-Hf Alloys

attributed to the 4 w/o additional solute concentration of the alloys. The Ta-11.2W-2.8Hf alloy displayed hardness values which were inconsistent with respect to the other Ta-W-Hf alloys. This anomalous behavior is probably a result of the tungsten segregation which was found to exist in this heat of material. Homogenous material of this composition would be expected to behave in a manner similar to the other three alloys.

Summary

Increasing the total solute constituent of T-111 from 10 to 14 w/o greatly improved high temperature mechanical properties. However, alloys containing 6 w/o Hf were subject to hot cracking during solidification in both the primary consolidation and in the welding stages. Increasing the total alloy content to 14 w/o also raised the ductile to brittle transition temperature for welded material. The alloy with a 4:1 W-Hf ratio was least affected in this respect and represents a minimum in the transition temperature for weld material tested over a 2T bend radius. The results of this survey indicate the high temperature properties of T-111 can be improved by increasing the total solute concentration from 10 to 14 w/o while maintaining a 4:1 W-Hf ratio at the sacrifice of some weldability.

INVESTIGATION OF ADDITIONAL TANTALUM ALLOYS

To assess the potential of several other interesting tantalum base alloys as high temperature structural materials, additional alloys were prepared and evaluated using the same criteria developed for the Ta-W-Hf alloy investigation. The alloys were selected to evaluate the effect of rhenium on the properties of Ta-W and Ta-W-Hf alloys. Work in an earlier phase of this program[1] had indicated that rhenium additions were potent strengtheners in tantalum. A Ta-W-Zr alloy was prepared to evaluate the possibility of substituting zirconium for hafnium in T-111. Tensile, stress-rupture, and weldability data were obtained when the yield of useful material permitted.

Melting and Processing

The alloys listed in Table 21 were prepared as button ingots and processed to sheet in the same manner as the Ta-W-Hf alloys previously described.

TABLE 21 - Chemical Analysis of Ta-Base Alloys

Heat No.	Nominal Composition (w/o)	Chemical Analysis (w/o)		
		C	O	N
TBM-13	Ta-4W-4Re	0.0058	0.0019	0.0023
TBM-19	Ta-4W-4Hf-4Re	0.0088	0.0410	0.0090
Ta-16	Ta-4W-4Hf-4V	--	--	--
TBM-1	Ta-8W-1Zr	0.0022	0.0011	0.0006

Heat Ta-16 was originally made as a single AC arc-melted ingot. This ingot was produced as part of a previous program[1]. Sheet was produced from the as-cast ingot by aluminide coating ingot slices 1" thick by 1-3/4" in diameter. Two such slices were coated and upset forged 50%. The forged slabs were conditioned, recrystallized one hour at 3000°F and cold rolled to 0.055" sheet.

Heats TBM-1, 13, and 19 were produced as button ingots. Starting material for heat TBM-19 was obtained from unused electrode stock from a previous program[1]. Heats TBM-1 and 13 were made from the same tantalum starting stock used for all other non-consumably arc melted buttons.

Heat TBM-19 (Ta-4W-4Hf-4Re) displayed severe cracking on solidification. However, some sound material was salvaged from the button ingot. This material was aluminide coated and forged successfully 50% at 2200°F. The forged slab was conditioned and recrystallized. During cold rolling, a severe longitudinal split occurred and rendered the material useless for further evaluation.

Heats TBM-1, 13 and Ta-16 were successfully rolled to 0.050" thick sheet. These alloys were evaluated for tensile properties and weldability.

Low and High Temperature Tensile Properties

The low and high temperature properties of heats TBM-1, 13, and Ta-16 are given in Table 22. TBM-1, Ta-8W-1Zr, was T-111 with the Hf replaced with an equivalent amount of Zr, on an atomic per cent basis. The tensile properties were comparable to and in some cases superior to the properties of low interstitial T-111 at all test temperatures. The ductility, as represented by elongation at fracture was also comparable except at the 3000°F test temperature. The Zr containing alloy displayed an elongation of 65 per cent, which was approximately twice that of T-111 at the same temperature.

The other alloys TBM-13, Ta-4W-4Re, and TA-16, Ta-4W-4Hf-4V, exhibited tensile properties which were also equivalent to or better than those for low interstitial T-111. At -320°F, however, both of these alloys displayed a trend toward brittle behavior. The Ta-4W-4Re alloy had an elongation of only 9% as compared to 30% for T-111. The Ta-4W-4Hf-4V alloy failed in the fillet of the test specimen with a brittle fracture. The addition of Re and V to Ta base alloys containing W appear to be potent strengtheners for short time tensile properties at elevated temperature and also appear to adversely affect low temperature ductility.

TABLE 22 - Low and High Temperature Tensile Properties of Ta-Base Alloys

Material	Temperature (°F)	Ultimate Tensile Strength (ksi)	Yield Strength 0.2% Offset (ksi)	Elongation (%)	Remarks
TBM-1	-320	150.2	141.3	30	Material reduced 80%.
Ta-8W-1Zr	75	93.4	82.8	33	Recrystallized 1 hr. at
	2400	42.3	28.6	33	3000°F
	3000	18.7	18.2	65	
TBM-13	-320	171.5	161.7	9	Material reduced 70%.
Ta-4W-4Re	RT	108.8	98.0	25	Recrystallized 1 hr. at
	2400	37.3	28.0	34	3000°F
	3000	21.8	17.4	38	
Ta-16	-320*	--	--	--	Material reduced 70%.
Ta-4W-4Hf-4V	RT	95.3	83.5	30	Recrystallized 1 hr. at
	2400	52.5	39.4	17	3000°F
	3000	17.8	17.1	26	

Strain rate at low temperature 0.005 in/in/min
Strain rate at high temperature 0.05 in/in/min

*Specimen failed in fillet with a brittle fracture.

Stress Rupture Properties. Sufficient sheet material was available from heats TBM-1 (Ta-8W-1Zr) and Ta-16, (Ta-4W-4Hf-4V) to permit evaluation of stress rupture properties. Tests were conducted at 2400°F in a vacuum of 5×10^{-5} Torr or less. The gauge length of the specimens was wrapped in Ta-foil prior to testing. Results are listed in Table 23.

TABLE 23 - 2400°F Stress Rupture Properties of Ta-Base Alloys

Material	Stress (ksi)	Minimum Creep Rate (%/hr)	Transition Time (hrs)	Rupture Time (hrs)	Elongation (%)	Remarks
TBM-1	30	5.7	0.05	0.38	15	Material reduced 80%
Ta-8W-1Zr	20	0.13	4.1	28.0	28	Recrystallized 1 hr. at 3000°F
Ta-16	40	9.7	1.2	1.5	25	Material reduced 70%
Ta-4W-4Hf-4V	30	0.46	5.5	30.2	36	Recrystallized 1 hr. at 3000°F

The stress for one hour rupture life for heat TBM-1 (Ta-8W-1Zr) as derived from the data in Table 23 was 28,000 psi as compared to 35,500 psi (Table 22) for low interstitial T-111. The stress for ten hour rupture life was 22,000 psi compared to 26,000 psi for the low interstitial T-111. The lower stress rupture properties for the Ta-8W-1Zr alloy were probably due to the replacement of Hf with the lower melting Zr.

The Ta-4W-4Hf-4V alloy which had a 42,500 psi stress for one hour rupture life displayed a significant improvement over the 33,500 psi stress for low interstitial T-111. For ten hour life, however, the difference in stress for the two alloys was only 2000 psi, 28,000 for the Ta-4W-4Hf-4V and 26,000 psi for low interstitial T-111. These data indicate that the addition of V to a Ta-base alloy greatly improves the short time properties, but as stress levels were reduced and test times increased the effect of the V was greatly reduced.

Welding Characteristics

Of the three alloys which were reduced to sheet, the Ta-8W-1Zr, Ta-4W-4Re, and the Ta-4W-4Hf-4V, only the Ta-4W-4Re welded without difficulty. The Ta-8W-1Zr and the Ta-4 W-4Hf-4V alloys were susceptible to hot tearing. In the Ta-4W-4Hf-4V alloy, the hot crack propagated the length of weld, thus making further evaluation impossible. Attempts to repair the hot crack in the Ta-8W-1Zr by rewelding were successful, allowing further evaluation.

Bend tests were conducted on as-welded material of the heats TBM-1 and TBM-13 using 1/4" wide by 1-1/8" long specimens with the weld running parallel to the bend axis. Results are listed in Table 24. The determination of weld bend transition temperature for 2T and 4T conditions was limited by the material that was available. A deflection rate of 1" per minute was used here also in accordance with the procedures established for evaluating the T-111 alloys.

TABLE 24 - Weld Bend Ductility of Ta-Base Alloys

Material	Remarks
TBM-1 Ta-8W-1Zr	Ductile over 2T at 150°F Ductile over 4T at room temperature
TBM-13 Ta-4W-4Re	Brittle over 2T at room temperature

It appears that in the case of each alloy the 2T bend transition temperature is above room temperature. The addition of as little as 1 per cent Zr, and Re in amounts greater than 2 per cent have a pronounced adverse effect on the weldability of Ta-base alloys containing W.

Summary

It appears that the replacement of Hf in low interstitial T-111 with an equivalent atomic per cent of Zr does not adversely affect tensile properties or fabricability. However, the lower melting Zr apparently induces hot tearing during welding and also raises the weld bend transition temperature of the alloy to above room temperature. The presence of Zr in T-111 in place of Hf also reduces the stress for one and ten hour rupture life and undoubtedly would have an adverse effect on longer time properties.

The addition of Re in amounts to 4 w/o to W containing Ta-base alloys appears to benefit short time tensile properties, although a significant reduction in low temperature ductility was noted. Re also significantly raises the weld bend transition temperature of W containing Ta-base alloys. It appears then that the most practical Ta-base alloys will be found in systems whose major constituents are W, Hf, and possibly Mo with lesser quantities of elements such as Re and possibly Os. Removing the criterion of weldability expands greatly the field of potential Ta-base alloys which can be produced and fabricated to useful shapes. However, weldability ranks with elevated temperature strength in importance in most applications.

SUMMARY AND RECOMMENDATIONS

The major objectives of this program were:

1. To investigate the properties of T-111 in detail

2. Establish the effects of interstitial elements on the properties of T-111

3. Optimize the tungsten and hafnium concentration in T-111

The properties of low interstitial T-111 have been outlined in some detail. In general, the alloy can be melted readily by using either AC or DC power, although initial melting by AC power is preferred. Primary breakdown of the as-cast ingot can be accomplished by either extrusion or by side and upset forging. Forging can be accomplished at 2200°F and results in a high yield of useful material with the use of an Al-Si coating to prevent contamination. After the material has been conditioned and recrystallized it can be cold rolled to high quality thin gauge sheet. Recrystallized material provides optimum mechanical properties over the entire temperature range of interest. In this condition the material is ductile in tension to -453°F, with excellent tensile properties in the 2500 to 3500°F temperature range. The material in the as-welded condition is ductile over a 2T bend radius at a temperature of -320°F for either stress-relieved or recrystallized base material. The addition of up to 0.02 w/o of carbon, oxygen or nitrogen has no appreciable effect on fabricability or weldability. Carbon and nitrogen, however, exert a significant effect on elevated temperature tensile properties. Oxygen appears to have little or no effect.

Increasing the individual interstitial level to 0.05 w/o does not significantly improve short time tensile properties, while higher carbon raises the weld bend transition temperature and higher oxygen and nitrogen content appears to adversely affect fabricability, particularly forgeability.

Solute concentration in T-111 was increased from 10 w/o total addition to 14 w/o at W/Hf ratios of 1.3 to 6:1 without adversely affecting fabricability and improving elevated temperature tensile properties. Weld bend ductility as expressed by transition from ductile to brittle behavior appears to be influenced by the tungsten-hafnium ratio, with a minimum in transition temperature occurring at a ratio of 4 to 1.

It appears from the data produced in this program that the properties of T-111 can be improved considerably without sacrificing its excellent fabricability or weldability. Improvement can be made by:

1. Increasing the amount of carbon and/or nitrogen in the basic T-111 composition, Ta-8W-2Hf.

2. Increasing the solute content to a higher level, 14 w/o, maintaining a tungsten-hafnium ratio of 4 to 1, while keeping total interstitial content at a low level.

3. Increasing the solute concentration of an intermediate level such as 12 w/o, maintaining the 4 to 1 tungsten-hafnium ratio and including 0.01 w/o carbon.

The latter recommendation is proposed as the best of the three alternatives, since it offers the greatest potential for further improvement in properties through optimization in processing and heat treatment to exploit the solute-interstitial interaction. Carbon can also be added easily in controlled amounts whereas additions of nitrogen though desirable would be difficult to control. The results of this program suggest the alloy Ta-9.6W-2.4Hf-0.01C as a composition worthy of further evaluation.

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